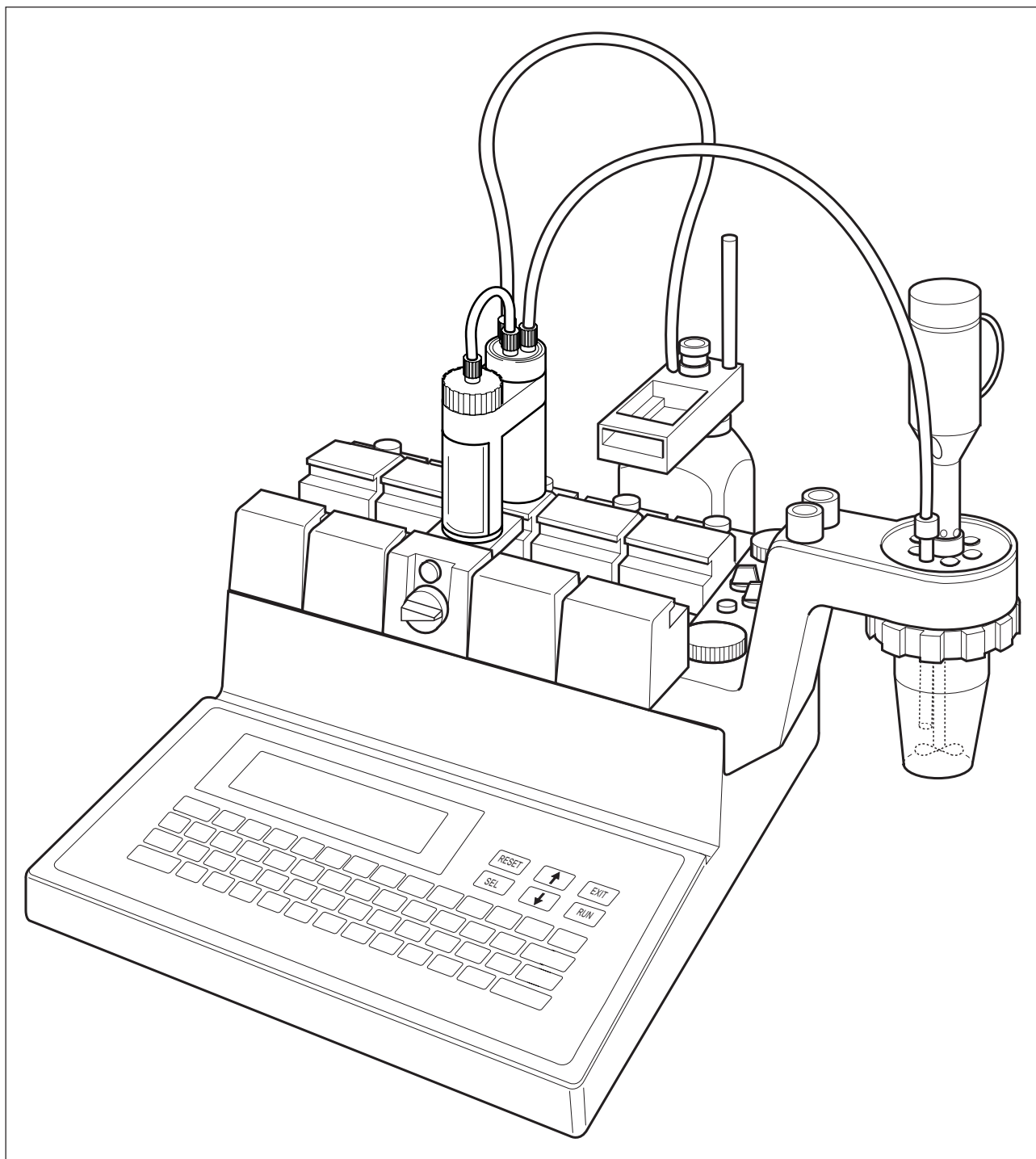


Operating Instructions

METTLER TOLEDO

DL77 / DL70ES / DL67 Titrators



1. INSTALLATION

2. EDITOR

3. ANALYSIS

4. AUXILIARY FUNCTIONS

5. DOCUMENTATION

6. USER LEVEL

7. REMOTE CONTROL

8. Designations – Explanations – Examples

9. Error messages and malfunctions

10. Applications

11. Installation instructions, Technical data, Accessories

12. Index, Certificates & Declarations

Safety measures

The titrators have been tested for the experiments and intended purposes documented in the Tutorial and these Operating Instructions. However, this does not absolve you from the responsibility of performing your own tests of the products supplied by us regarding their suitability for the methods and purposes you intend to use them for. You should therefore observe the following safety measures.

Measures for your protection



Risk of electric shock

- Ensure that you plug the power cable supplied into a receptacle outlet that is grounded! In the absence of grounding, a technical fault could be lethal.
- Switch the instrument off and disconnect the power cable before you open the housing or change blown fuses! An electric shock could be lethal.



Risk of explosion

- Never work in an environment subject to explosion hazards! The housing of the instrument is not gas tight (explosion hazard due to spark formation, corrosion caused by the ingress of gases).



Risk of corrosion

- Always test the titration vessel for firm seating in the titration head! If it falls off, you could injure yourself if working with toxic titrants and solvents or strong acids or bases.
- When using chemicals and solvents, comply with the instructions of the producer and the general lab safety rules! Additional safety precautions for Karl Fischer titrations are described in Section 10.2.

Measures for operational safety



Caution

- Check the set operating voltage before you switch on the titrator (see Section 11.1.5)! The instrument will be damaged if the operating voltage does not match the line voltage.
- Use only fuses of the specified type if you need to change them!– Have the instrument serviced only by METTLER TOLEDO Service!
- Always wipe off splashed liquids immediately! The instrument is not waterproof.
- Exclude the following environmental influences:
 - powerful vibrations,
 - direct sunlight,
 - atmospheric humidity greater than 80%,
 - temperatures below 5 °C and above 40 °C,
 - powerful electric or magnetic fields!

Introduction

The DL77, DL70 ES, and DL67 METTLER titrators are microprocessor-controlled analytical instruments that provide accurate and reproducible results thanks to their built-in intelligence.

With these titrators, you can perform end point, equivalence point and pH-stat titrations, measure pH/mV and temperature, and determine TAN/TBN and p/m values. With the aid of a polarization current source, you can determine water contents by the Karl Fischer method ($>2 \text{ mg H}_2\text{O/sample}$). You can perform conductivity measurements and conductometric titrations with an appropriate non-Mettler unit equipped with an analog output.

All titrators have a maximum of four inputs for electrodes, two for temperature sensors and three 24-V outputs for stirrer, pump or valve attachments. They have a maximum of four RS232C/CL interfaces, enabling you to connect a series of peripheral instruments:

- an attached METTLER balance transfers the sample weight automatically,
- a printer records the desired results,
- a color terminal serves as a second display and can be used for remote control, or a PC/robot can interchange data with the titrator, and
- with an attached METTLER Sample Changer, each titrator is turned into a titration system for the automatic analysis of whole series of samples.

How the three titrators differ from each other

DL77: Two titrations can be executed simultaneously and two sample changers can be connected. Neither the DL70ES nor the DL67 allow this.

DL77/ These titrators can control a maximum of four burette drives, the DL67 two.

DL70ES: These instruments know the most important titrants and all METTLER sensors, whereas the DL67 knows one titrant and one sensor.

With both titrators a maximum of 10 titration methods can be entered in a list and processed in succession. With the DL67 one method can be entered at a time.

While a titration is running, you can develop and save a new method, or you can enter sample data for the next titration method. This is not possible with the DL67.

What information will you find where?

1. The **TUTORIAL**, provided with the standard equipment, will help you to overcome any inhibitions you may have with regard to the new instrument. You will get to know the function keys, the keypad and the display. Using a stored method for an acid-base titration, you will perform your first analysis.
2. These **Operating Instructions** provide a complete description of the concept and operating characteristics of the three titrators.
The operating concept and a compilation of the key combinations follow this introduction.
3. The **RS232C Interface Description**, i.e., a detailed description of the communication between titrator and computer, is provided with the standard equipment (since June, 1999).

Note: These Operating Instructions apply to software version 3.0 or 3.1.

The organization of the Operating Instructions

The organization is based on a modular principle. This allows supplementation or the interchange of individual sections or pages: new text sections carry the date of issue (in the footer on every page).

The operating concept of the titrator

The operation of the titrator is menu driven. What does this mean?

The titrator handles various tasks:

It stores, for instance, titrant names with the corresponding concentrations; it knows that it must terminate the titration after an equivalence point has been found; it rinses burettes and can provide records of stored data on an attached printer.

We call the listing of these various tasks a menu. Each task is subdivided further. If you select a task from the main menu you are shown a new set of tasks – the submenu – from which you can select another task. We refer to this as a menu tree when the main menu branches into submenus and these in turn into additional submenus.






Rapid menu change with key combinations

In the Tutorial, you have already become acquainted with several key combinations which you can employ to obtain a certain menu immediately. Using these keys, you can reduce the number of keystrokes before and during the titrations considerably. The key combinations consist of the index key and a letter key which must be pressed simultaneously. Key combinations exist to

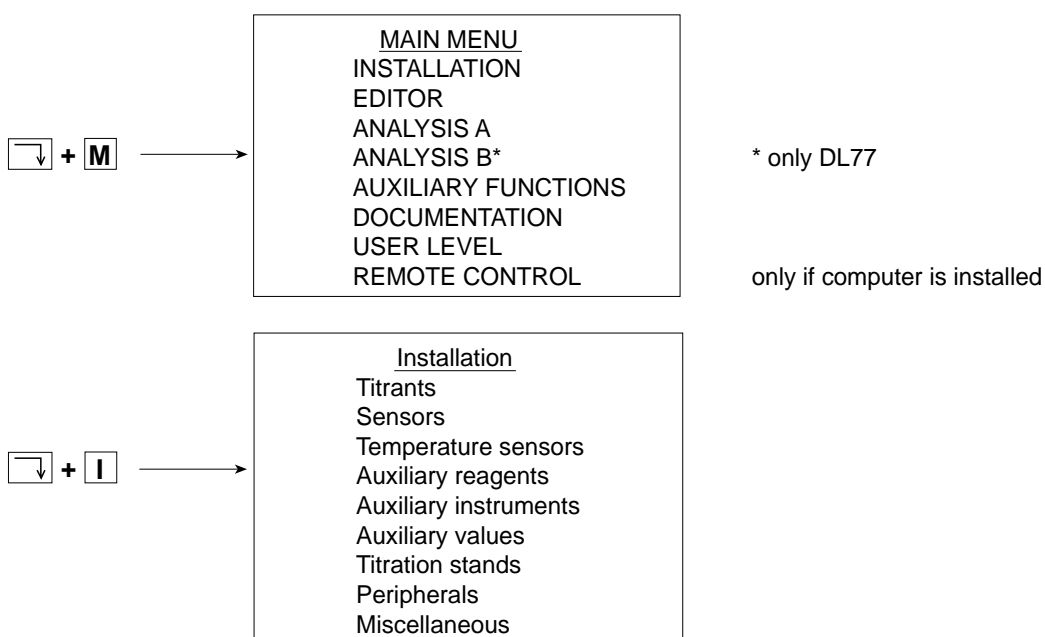
- jump from a submenu into the main menu or from a submenu into that of another branch, or to
- trigger commands for the burette or the printer (see below)








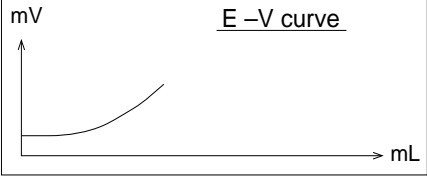
If you press a key combination in a submenu, the DL70 **stores all changes** that you have made up to this point in the menu. The selector bar can be on **any line** of the menu.

Key combinations for commands

 + U	Rinse burette (see Section 4.1.1)
 + Y	Rinse tip (see Section 4.1.2)
 + L	Trigger line feed on the printer (see Section 1.8.1)
 + P	Trigger form feed on the printer (see Section 1.8.1)
 + C	The current display is printed (copied!)

Key combinations for menu change



 + E	<div>Editor Method ID METTLER methods User methods New method</div>													
 + A	<div>MethodMethod Add*A</div>	*DL67: Load A: only DL77												
 + B	<div>MethodMethod AddB</div>	only DL77												
 + F	<div>Auxiliary functions 1 Burette 2 Stirrer 3 Sensor 4 Temperature 5 Sample changer 6 Auxiliary instrument 7 Calibration temperature sensors 8 Offset adjustment sensor inputs</div>	only if sample changer is installed only if temperature option has been built in												
 + D	<div>Documentation Print Data transfer Memory copy</div>													
 + S	<table><tr><th>No.</th><th>ID2</th><th>Wt./vol.</th></tr><tr><td>1/1</td><td></td><td>0.0000 g</td></tr><tr><td>1/2</td><td></td><td>0.0000 g</td></tr><tr><td>1/3</td><td></td><td>0.0000 g</td></tr></table>	No.	ID2	Wt./vol.	1/1		0.0000 g	1/2		0.0000 g	1/3		0.0000 g	Sample data list for the current method (example)
No.	ID2	Wt./vol.												
1/1		0.0000 g												
1/2		0.0000 g												
1/3		0.0000 g												
 + T	<div><div>E –V curve</div></div>	Display of the method function for the current method (Titration function active)												

Contents	Page
1. INSTALLATION	1-3
1.1 Titrants	1-4
1.1.1 Delete	1-4
1.1.2 Modify	1-4
1.1.3 Add	1-8
1.2 Sensors	1-9
1.2.1 Delete	1-10
1.2.2 Modify	1-10
1.2.3 Add	1-14
1.3 Temperature sensors	1-15
1.4 Auxiliary reagents	1-17
1.4.1 Delete	1-17
1.4.2 Modify	1-17
1.4.3 Add	1-18
1.5 Auxiliary instruments	1-20
1.5.1 Delete	1-20
1.5.2 Modify	1-20
1.5.3 Add	1-21
1.6 Auxiliary values	1-23
1.7 Titration stands	1-25
1.8 Peripherals	1-28
1.8.1 Printer	1-28
1.8.2 Balance	1-30
1.8.3 System	1-32
1.8.4 Sample changer	1-35

	Page
1.9 Miscellaneous	1-36
1.9.1 Format date/time	1-36
1.9.2 Enter date/time	1-36
1.9.3 Language	1-37
1.9.4 Record header	1-37
1.9.5 Titrator ID	1-37
1.9.6 Routine level	1-38
1.9.7 Audio signal	1-39
1.9.8 Analysis parameters	1-40

1. INSTALLATION

In order to perform titrations the titrator must be acquainted with the titrants and their concentration, the sensors with their possible unit of measurement, and the solvents that it can dispense by means of pumps. It must know the burette drive on which the burette is located, the input to which the sensor is connected and what output carries the stirrer. It needs the names of the attached units such as a balance or printer in order to transfer data. In this menu you enter and store the names of all chemical and mechanical resources: you **install** them. The most common titrants, solvents and all METTLER sensors are already installed in the titrator. Not only can you delete these **resources** or modify their parameters, but you also have the possibility to install new ones.

List of resources

Titrants
Sensors
Temperature sensors
Auxiliary reagents
Auxiliary instruments
Auxiliary values
Titration stands
Peripherals
Miscellaneous

Caution: All resources needed for the **METTLER methods** stored in the application data base are installed accordingly. If you delete one of these, the titrator will wait until start of titration of a METTLER method before outputting the error message that the resource is not installed.

DL67: Only the titrant and the sensor for METTLER method M001 are stored in the DL67.

1.1 Titrants

When you select this menu you receive the installed titrants with the specified parameters concentration and burette drive. (You will find the menu tree depicted at the end of Section 1.1.2.)

NaOH	0.1 mol/L	Drive 3
HCl	0.1 mol/L	Drive 3
HClO ₄	0.1 mol/L	Drive 3
etc...		

DL67: The DL67 only has stored the titrant NaOH. You can, however, add titrants to the list (see Section 1.1.3).

If you select, for instance, NaOH the list will be masked on the right by a selection menu containing the following commands:

Delete
Modify
Add

1.1.1 Delete

Position the selector bar on this command and confirm with **RUN**. The mask of the selection menu disappears, the titrant NaOH is deleted.

Note: You can also delete a titrant directly from the list by pressing the <-> (minus) key. You can reinstall NaOH with the command **Add** (see Section 1.1.3).

1.1.2 Modify

If you select this command you are shown the parameter mask of the titrant (see next page). If you move the selector bar to one of these parameters you can modify its name or value.

Note: a. Default values are stored in the titrator for all parameters, e.g. for the concentration of the titrant 0.1 mol/L, for the titer 1.0.

- These values will be overwritten once you have entered new ones.
- If you want to modify only one digit of a value, you must first indicate the digit with the cursor (with → or ←) prior to entering the new one.

Note: b. With many parameters the titrator has selection or recommendation menus in which you need only select the values or names. If a parameter has one of these menus, each time it is selected an arrow symbol (➡) appears at the extreme right in the middle of the display. You can either accept the value or name following the parameter or:

in the case of **Recommendation**,

- overwrite directly with a new entry or
- press **SEL** to select a new value or name from the recommendation menu that appears.

in the case of **Selection**,

- press **SEL** to select a new value or name from the selection menu that appears. You cannot enter the value or name yourself.

If only two names or values are possible for a parameter, these are toggled automatically with **SEL**.

Name	NaOH
Concentration [mol/L]	0.1
Titer	1.0
Burette volume	10 mL
Burette drive	Drive 3
Date/time	(e.g.: 07-06-1992 12:20)

1. You can overwrite the name or press **SEL** to call up the recommendation menu:

Recommendation menu: You can select a new titrant from the recommendation menu. Position the selector bar on, for instance, **HCl** and confirm with **RUN**. The recommendation menu disappears and **HCl** follows **Name**.

2. Enter the concentration [mol/L]
3. Enter the titer only if you know its value. If you determine the titer of the titrant with the titrator, its value is entered here together with the date automatically (see *Titer* function, Section 2.3.16).
4. Select the burette volume from the selection menu:
Selection menu: You press **SEL**, position the selector bar on, for instance, **5 mL** in the selection menu and confirm with **RUN**. The selection menu disappears and **5 mL** now follows **burette volume**.
5. Select the burette drive from the selection menu.

DL67: It is only possible to select one of two burette drives for this titrator. If more than two drives have been installed, the titrator will recognize each of the first two (counting from left to right).

6. You can neither delete nor overwrite the date. It refers to the titer determination of the titrant using the method function *Titer* and is entered here together with the time automatically (see *Titer* function, Section 2.3.16).

Note: As soon as you change a titrant parameter, the date and time are deleted.

The titrant with the modified parameters is installed when you quit the parameter mask with **EXIT**. Here, the selector bar can be positioned on any parameter, but not on the title line. If you confirm the title line with **EXIT** the following selection menu appears:

Save modifications?

Yes

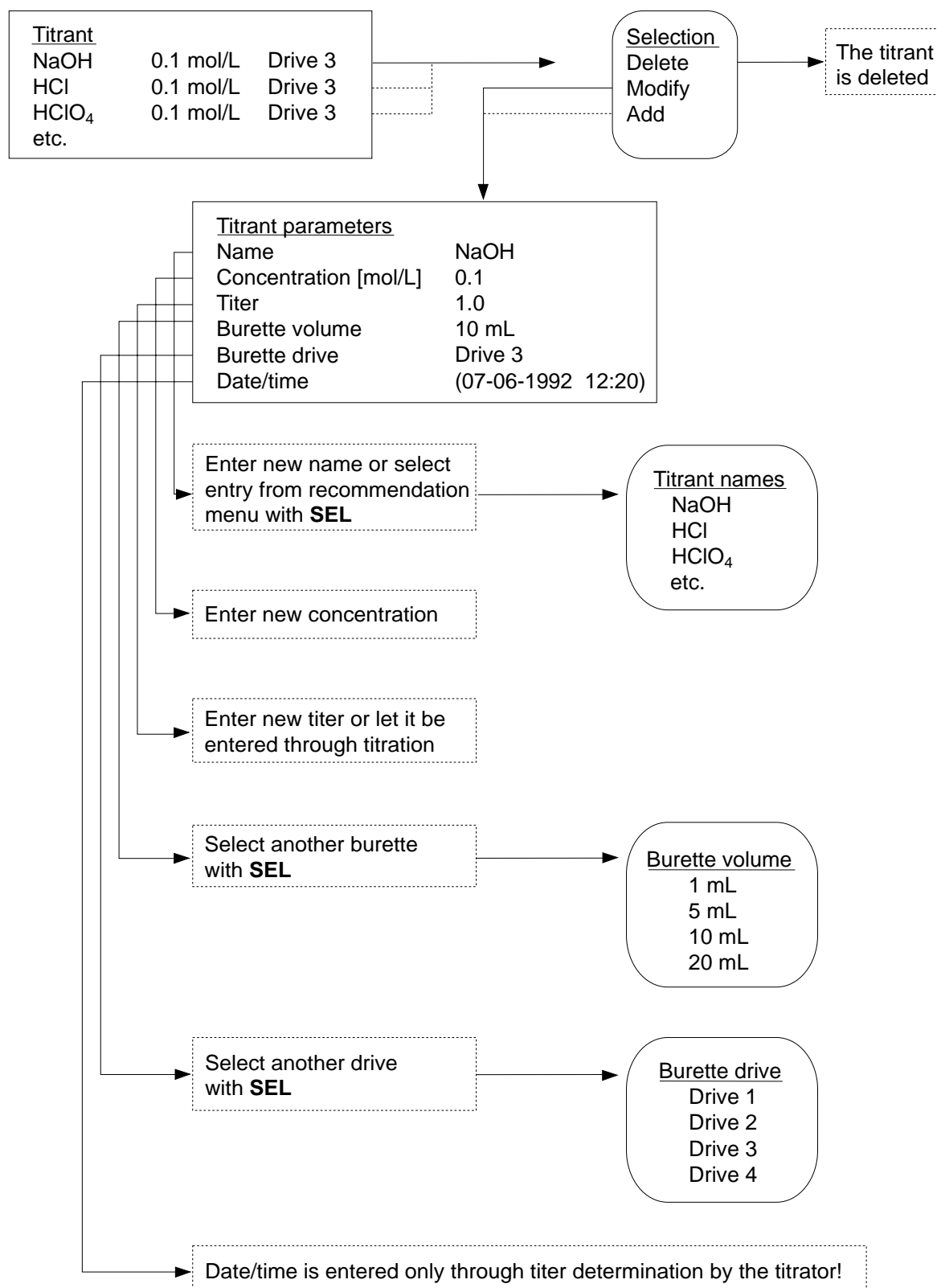
No

If you confirm "Yes" with **RUN**, the titrator stores the modified values or names.

If you confirm "No" with **RUN**, the old values remain in force.

Note: If you press a **key combination** (<index + letter>) to quit the Installation menu, the modified parameters are stored automatically if the selector bar is positioned on a parameter line. If it is positioned on the title line, the selection menu "Save modifications?" appears again (see above).

Menu tree



1.1.3 Add

Select this command if you wish to add a titrant to the titrant list, for instance NaOH of concentration 1.0 mol/L or with a different burette volume, or a new titrant. You can also add a titrant directly to the list by pressing the <+> (plus) key. You are always shown the following mask:

Name	NaOH
Concentration [mol/L]	0.1
Titer	1.0
Burette volume	10 mL
Burette drive	Drive 3
Date/time	00-00-0000 00:00

1. Select the titrant from the recommendation menu or enter the one you wish to install.

Note: If you install, for example, several NaOH solutions of the same concentration, you must provide each name with a different flag to allow the titrator to distinguish between them, e.g. NaOH/1.

2. Enter the concentration [mol/L].
3. Enter the titer only if you know its value. If you determine the titer with the titrator, its value together with the date is entered here automatically (see *Titer* function, Section 2.3.16).
4. Select the burette volume from the selection menu.
5. Select the burette drive from the selection menu.
6. The date together with the time for the newly installed titrant is entered here automatically only after the titer has been determined (see *Titer* function, Section 2.3.16).

The titrant with the appropriate parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

1.2 Sensors

When you select this menu you are shown the installed sensors with the specified parameters unit of measurement and the sensor input. (You will find the menu tree depicted at the end of Section 1.2.2.)

DG111-SC	pH	Sensor 1	(Combined pH electrode – aqueous medium)
DG101-SC	pH	Sensor 1	(Combined pH electrode for small volumes in a small titration vessel – aqueous medium)
DG113-SC	mV	Sensor 1	(Combined glass electrode with movable sleeve frit – nonaqueous medium)
DG114-SC	mV	Sensor 1	(Combined glass electrode with movable sleeve frit – aqueous medium)
DG115-SC	mV	Sensor 1	(Combined glass electrode with sleeve frit – aqueous medium)
DM140-SC	mV	Sensor 2	(Combined platinum ring electrode – redox titrations)
DM141-SC	mV	Sensor 2	(Combined silver ring electrode – argentometry)
DM142	mV	Sensor 1	(Double-pin platinum electrode – voltametry)
DP550	% T	Sensor 2	(Phototrode – transmission measurements at 550 nm)
DP660	% T	Sensor 2	(Phototrode – transmission measurements at 660 nm)

DL67: Only sensor DG111-SC is stored in the DL67. You can, however, add sensors to the list (see Section 1.2.3).

Note: There is no need to **install** a reference electrode as it is part of the installed measuring electrode at **input sensor 1**. The input for the reference electrode is marked (see Section 11.1.4).

If you select DG111-SC, for instance, the list is masked on the right by a selection menu containing the following commands:

Delete
Modify
Add

1.2.1 Delete

Position the selector bar on this command and confirm with **RUN**. The mask of the selection menu disappears, the DG111-SC sensor is deleted.

Note: You can also delete a sensor directly from the list by pressing the <=> (minus) key. You can reinstall the DG111-SC with the **Add** command (see Section 1.2.3).

1.2.2 Modify

If you select this command you are shown the parameter mask of the sensor in which you can change the parameter values:

Name	DG111-SC
Unit of measurement	pH
Sensor input	Sensor 1
Zero point [Unit]	7.0
Slope [mV/Unit]	-59.16
Temperature [°C]	25.0
Date/time	(e.g.: 02-06-1992 10:15)

1. Select the sensor name from the recommendation menu or enter the one you wish to install in place of the DG111-SC sensor.
2. Select the unit of measurement suitable for the sensor from the selection menu. Within a method you can later again choose between the selected unit of measurement and "mV" in the functions **Measure**, **Titration** and **pH/mV-stat** (see Section 2.3.4/ 12/13).

- mV:** Either there is no other unit of measurement suitable for the sensor, or you require only mV.
If you select the unit mV for a sensor, the calibration parameters zero point, slope and temperature are ignored by the titrator.
- pH:** After you select pH you then enter the calibration parameters for a pH electrode or you let the titrator do it (see notes a. and b. on page 1-12).
- pM:** M represents any cation. After you select pM you then enter the calibration parameters for a pM electrode or you let the titrator do it (see notes a. and b. on page 1-12).
- pX:** X represents any anion. After you select pX you then enter the calibration parameters for a pX electrode or you let the titrator do it (see notes a. and b. on page 1-12).

- $\%T$: After you select $\%T$ you then enter the calibration parameters of a phototrode (unit of measurement: transmission, see *Operating Instructions "Phototrode"*).
- A: After you select A you then enter the calibration parameters of a phototrode (unit of measurement: transmission. The absorption ($A = -\log T$) is calculated, see *Operating Instructions "Phototrode"*).
- $\mu S/cm$: After you select $\mu S/cm$ you then enter the calibration parameters of the conductivity cell.
- mS/cm: After you select mS/cm you then enter the calibration parameters of the conductivity cell.

Note: Conductivity measurements and conductivity titrations can be performed using a conductometer equipped with an analog output.

3. Select the sensor input from the selection menu.

Notes: a. In the standard configuration of the titrator you have the sensor 1 and sensor 2 input available. You should select the sensor 1 input for all glass electrodes as this has the highest input resistance (see *Rear view of the titrator*, Section 11.1.4 and *Technical data*, Section 11.2.1).

- b. For technical reasons, only the "low resistance" sensor input 2 may be selected for phototrodes (see *Rear view of the titrator*, Section 11.1.4 and *Technical data*, Section 11.2.1).

Caution: The RS option has an additional "low resistance" sensor input, the temperature option an additional high resistance sensor input (see Sections 11.1.6 and 11.1.7). Depending on the installation manner, the "low resistance" input may, for example, be either sensor 3 or sensor 4! (See *Rear view of the titrator*, Section 11.1.4 and *Technical data*, Section 11.2.1).

4. Enter the zero point of the sensor:

The zero point of a sensor is the measured value at which it displays **0 mV potential** (zero point of electrode assembly).

- For the zero point of a pH electrode this is pH_0 with unit pH.
- For the zero point of an ion-selective electrode this is pM_0 with the unit pM, or pX_0 with the unit pX.
- The zero point of the phototrode is normally 0% T (100% T = 1000 mV).
- The zero point of a conductivity cell is normally 0 $\mu S/cm$ or 0 mS/cm.

5. Enter the slope of the sensor.

The slope of a sensor is the **potential change in mV per unit of measurement**.

- For the slope of a pH electrode the unit is: mV/pH.
- For the slope of an ion-selective electrode the unit is: mV/pM or mV/pX.
- The slope of a phototrode is normally 10 mV/ %T.
- For the slope of a conductivity cell the unit is: mV/ $\mu\text{S}\cdot\text{cm}^{-1}$ or mV/ $\text{mS}\cdot\text{cm}^{-1}$.

6. Enter the temperature: The actual calibration temperature is either

- defined by you when starting a calibration method (see Section 3.1) or
- measured automatically if you have attached and installed a temperature sensor (see Sections 1.3, 2.3.2 and 2.3.17).

7. You can neither delete nor overwrite the date. It refers to the calibration of the sensor using the method function *Calibration* and is entered here together with the time automatically (see *Calibration* function, Section 2.3.17).

Notes: a. The calibration parameter values of the titrator installed in the factory are theoretical values for a **new** sensor. You have to perform a calibration of your sensor if you wish to determine accurate values (see *Calibration* function, Section 2.3.17).

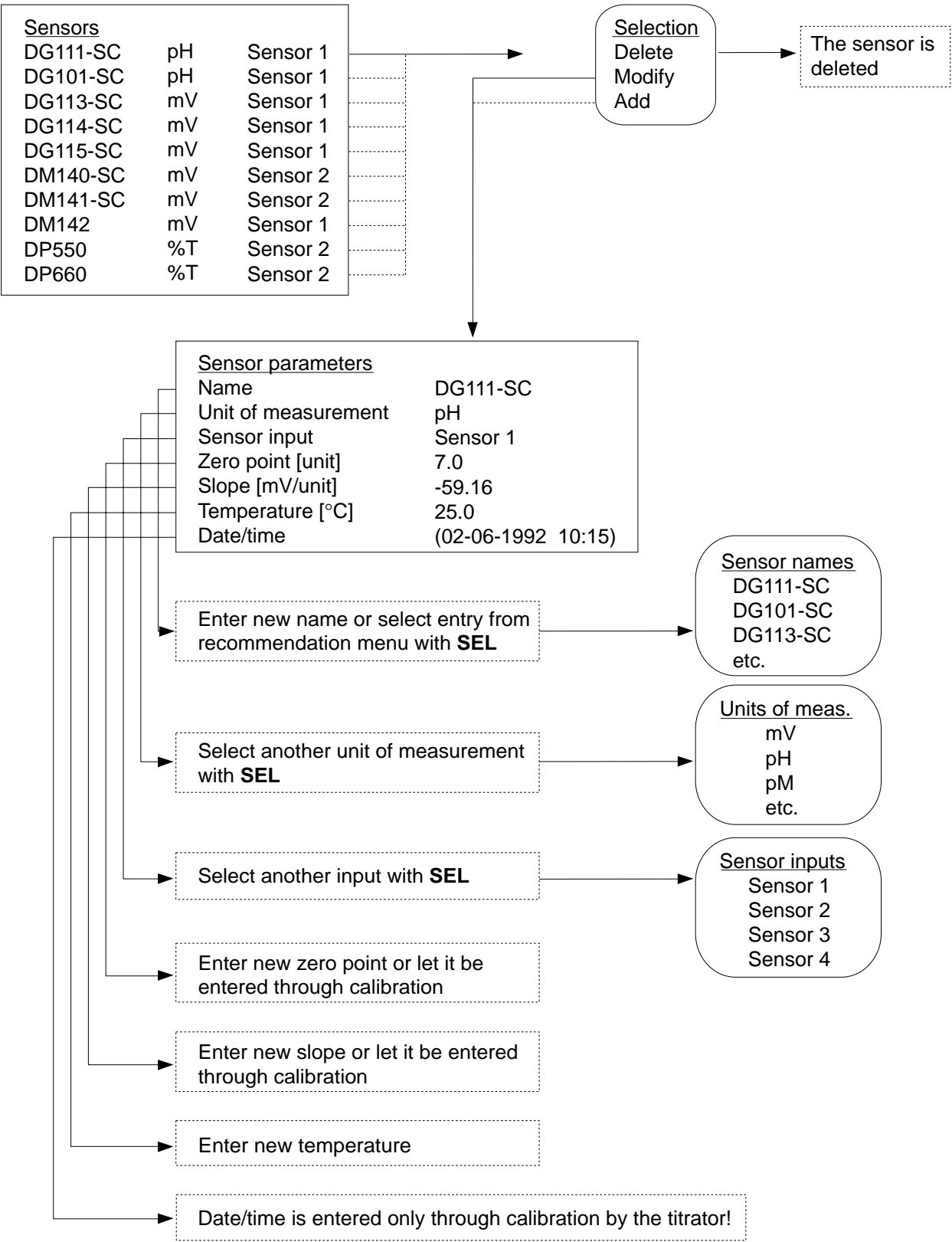
b. When a pH, pM or pX sensor is calibrated the calibration parameters (zero point, slope, temperature) are entered here automatically.

c. As soon as you change a sensor parameter, the date and time are deleted.

Caution: Do not transfer the calibration data obtained for glass electrodes attached to sensor input 1 (or for the temperature option sensor input) to the electrodes you attach to sensor input 2 (or the sensor input of the RS option)! Recalibrate these sensors to obtain correct values!

The sensor with the modified parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

Menu tree



1.2.3 Add

Select this command if you wish to add a sensor to the sensor list, for instance a DG111-SC sensor with different calibration parameters or a sensor not yet installed. You can also add a sensor directly to the list by pressing the <+> (plus) key. You are always shown the following mask:

Name	DG111-SC
Unit of measurement	mV
Sensor input	Sensor 1
Zero point [Unit]	7.0
Slope [mV/Unit]	-59.16
Temperature [°C]	25.0
Date/time	00-00-0000 00:00

1. Select the sensor name from the recommendation menu or enter the name of the sensor you wish to install.

Note: If you install, for example, several DG111-SC sensors, you must provide each name with a different flag to allow the titrator to distinguish between them, e.g. DG111/2.

2. Select the unit of measurement suitable for the sensor from the selection menu.
3. Select the sensor input from the selection menu.
4. Enter the zero point of the sensor.
5. Enter the slope of the sensor.
6. Enter the temperature.
(see explanation of the parameters and note under *Modify*).
7. The date together with the time for the newly installed sensor is entered here automatically only after the sensor has been calibrated (see *Calibration* function, Section 2.3.17).

The sensor with the appropriate parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

1.3 Temperature sensors

When you select this menu you are shown the installed temperature sensors with the specified parameters sensor type and sensor unit:

TEMP A	Pt100	Temp 1
TEMP B	Pt100	Temp 2
TEMP C	Pt1000	Temp 1
TEMP D	Pt1000	Temp 2

You can neither delete a temperature sensor nor add a new one to the list. You can, however, install several Pt100 or Pt1000 at the same temperature sensor input (Temp 1 or Temp 2), for example.

If you select TEMP A, for instance, you are shown the parameter mask of the Pt sensor in which you can change the parameter values or names:

Name	TEMP A
Sensor type	Pt100
Sensor input	Temp 1
Zero point [°C]	0.0
Date/Time	00-00-0000 00:00

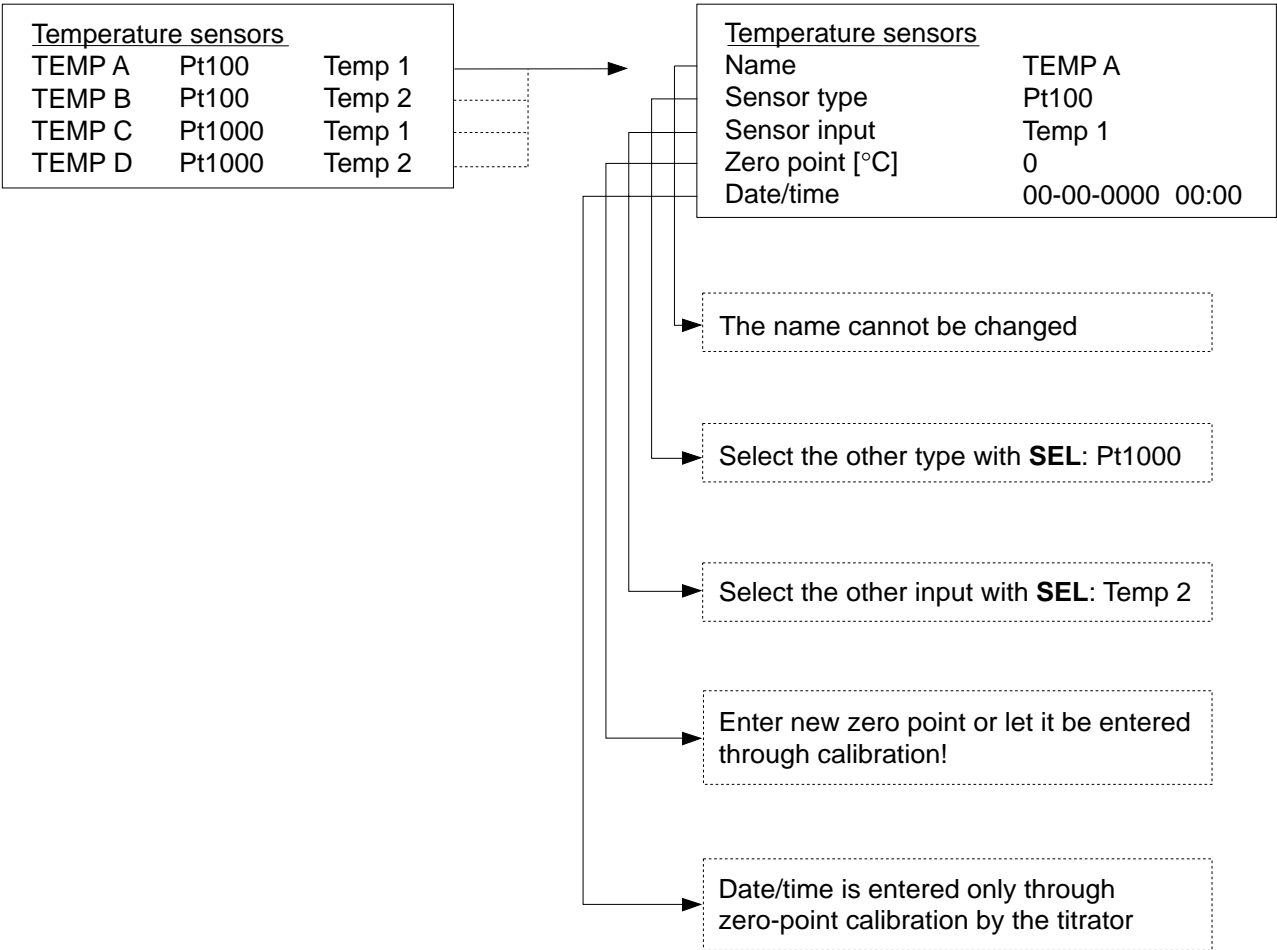
1. You can not change the sensor name: The line will be skipped when the selector bar is moved.
2. Select the sensor type with **SEL**: "Pt100" or "Pt1000" (see Section 11.1.7).
3. Select the sensor input with **SEL**: "Temp 1" or "Temp 2" (see Section 11.1.4).
4. Enter the zero point of the Pt sensor.
The zero point of a Pt sensor is the deviation of a measured value from a reference value, such as **0 °C** for an ice bath.
5. You can neither delete nor overwrite the date. It refers to the calibration of the Pt sensor using the auxiliary function *Calibration temperature sensors* and is entered here together with the time automatically (see Section 4.7).

Notes: a. The installed zero point is the theoretical value for a **new** Pt sensor. You have to perform a calibration of your sensor if you wish to determine an accurate value. When calibrating, the zero point is automatically entered here together with the date (see auxiliary function *Calibration temperature sensors*, Section 4.7).

- b. As soon as you change a parameter of the temperature sensor, the date and time are deleted.

The Pt sensor with the modified parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

Menu tree



1.4 Auxiliary reagents

When you select this menu you receive the installed auxiliary reagents with the specified parameters dispensing rate and auxiliary output. An auxiliary reagent is a solvent that is dispensed using a time-controlled device, e.g. a diaphragm pump or an electromagnetic valve. (You will find the representation of the menu tree at the end of Section 1.4.3).

H ₂ O	250 mL/min	Aux. 2
CH ₃ OH	250 mL/min	Aux. 2
CHCl ₃	250 mL/min	Aux. 2
etc.		

DL67: Only auxiliary reagent H₂O is stored in the DL67. You can, however, add auxiliary reagents to the the list (see Section 1.4.3).

If you select H₂O, for instance, the list is masked on the right by a selection menu containing the following commands:

Delete
Modify
Add

1.4.1 Delete

Position the selector bar on this command and confirm with **RUN**. The mask of the selection menu disappears, the auxiliary reagent H₂O is deleted.

Note: You can also delete an auxiliary reagent directly from the list by pressing the <--> (minus) key. You can reinstall H₂O with the command **Add** (see Section 1.4.3).

1.4.2 Modify

If you select this command you are shown the parameter mask of the auxiliary reagent in which you can change the parameter values:

Name	H ₂ O
Dispensing rate [mL/min]	250
Auxiliary output	Aux. 2

1. Select the name of the auxiliary reagent from the recommendation menu or enter the name of the reagent you wish to install in place of H₂O.

2. Enter the dispensing rate [mL/min] of the device.

Notes: a. The titrator uses the dispensing rate to calculate the volume to be dispensed for time-controlled pumps or electromagnetic valves (see functions **Pump** and **Rinse**, Sections 2.3.8 and 2.3.9).

- b. The dispensing rate of each device must be determined experimentally in advance for each solvent:
- Add the particular solvent using the dispensing device to a measuring cylinder within 1 minute (stopwatch) and note the volume.
 - Repeat this procedure for, e.g. periods of 20, 30 and 40 seconds.
 - Use the different volumes to calculate the mean value per minute and enter this value.

3. Select the auxiliary output from the selection menu.

Note: The titrator has three auxiliary outputs (Aux. 1, 2 and 3). One auxiliary output of the titrator is normally assigned to the stirrer, e.g. Aux. 1 (see Section 1.7). You should thus select only Aux. 2 and/or Aux. 3 for this auxiliary reagent (see *Technical data*, Section 11.2.2).

If you have attached the sample changer (ST20A or ST20), this unit offers you two additional outputs called "RINSE" and "DOSE".

The auxiliary reagent with the modified parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

1.4.3 Add

Select this command when you wish to add an auxiliary reagent to the list. You can also add an auxiliary reagent directly to the list by pressing the <+> (plus) key. You are always shown the following mask:

Name	H ₂ O
Dispensing rate [mL/min]	250
Auxiliary output	Aux.2

1. Select the name from the recommendation menu or enter the reagent you wish to install.

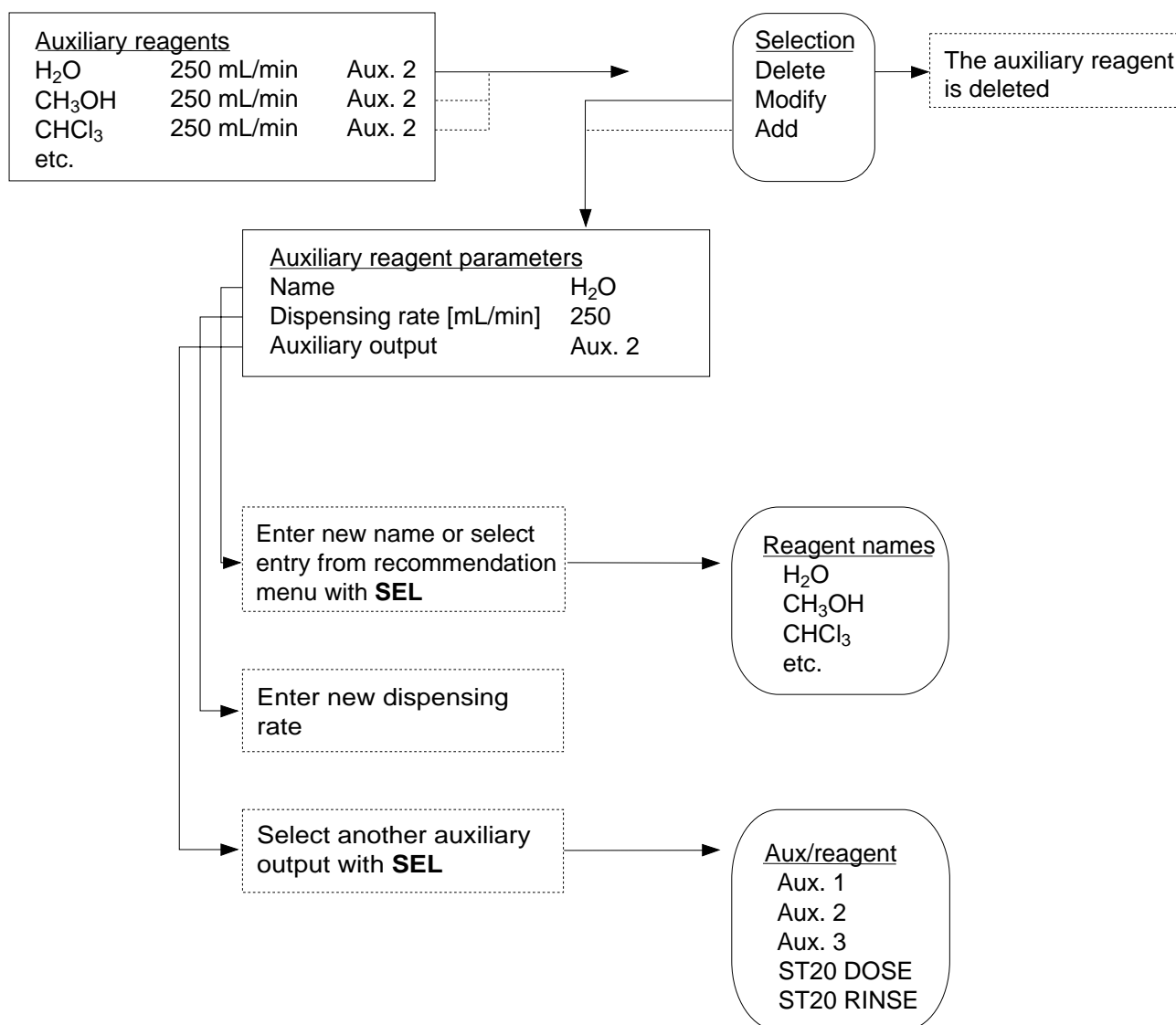
Note: If you install several auxiliary reagents with the same name, you must provide each one with a different flag to allow the titrator to distinguish between them, e.g. H₂O/2.

2. Enter the dispensing rate [mL/min] of the device.

3. Select the auxiliary output from the selection menu.

The auxiliary reagent with the appropriate parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

Menu tree



1.5 Auxiliary instruments

When you select this menu you are shown the installed auxiliary units with the specified parameter auxiliary output. Auxiliary instruments can be pumps, dispensers, valves or relays that have a 24 V connector. They are controlled by the titrator; the actual function of the units is unknown to the titrator.

Pump	Aux.3
Dispenser	Aux.3
Valve	Aux.3

If you select pump, for instance, the list is masked on the right by a selection menu containing the following commands:

Delete
Modify
Add

1.5.1 Delete

Position the selector bar on this command and confirm with **RUN**. The mask of the selection menu disappears, the auxiliary unit pump is deleted.

Note: You can also delete an auxiliary instrument directly from the list by pressing the <-> (minus) key. You can reinstall "Pump" with the command **Add** (see Section 1.5.3).

1.5.2 Modify

If you select this command you are shown the parameter mask of the auxiliary instrument in which you can change the parameter values:

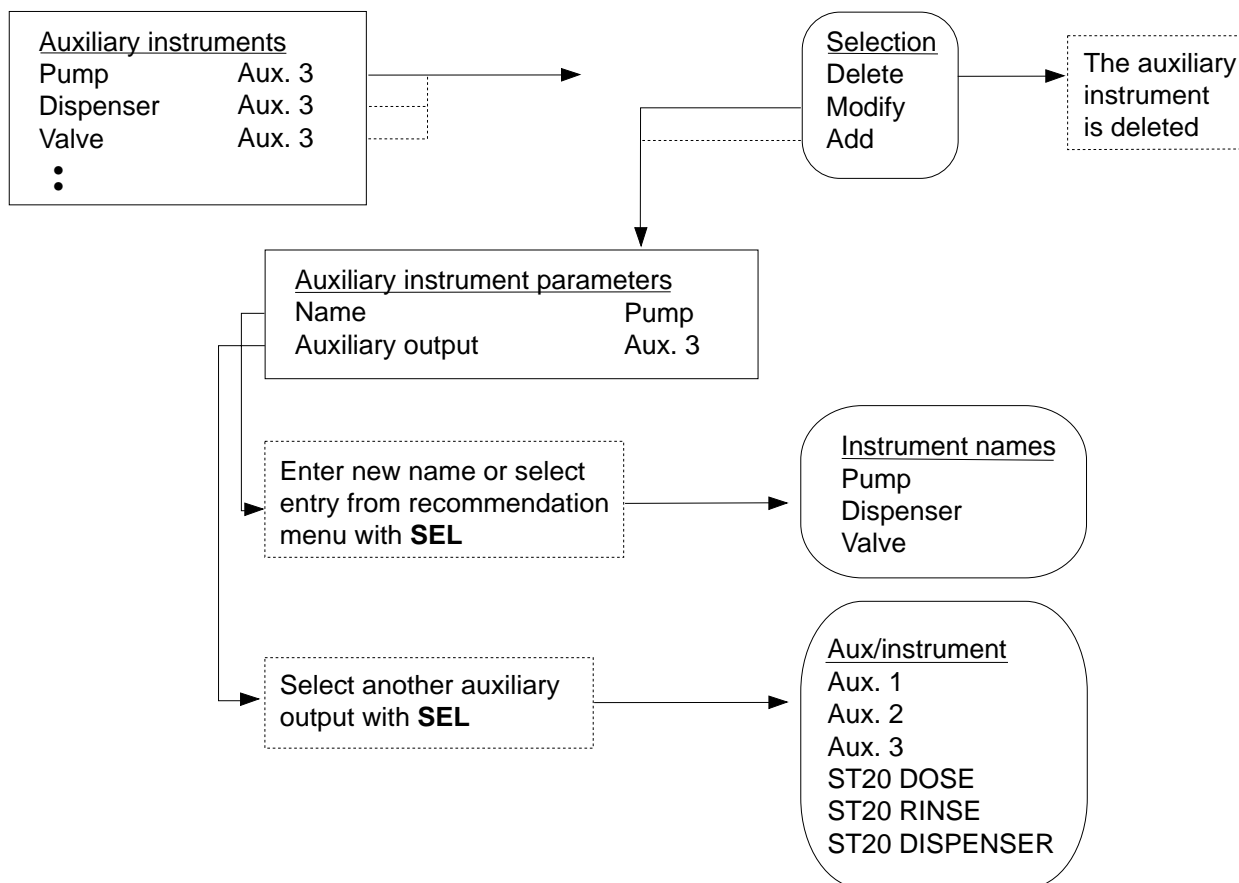
Name	Pump
Auxiliary output	Aux.3

1. Select the name from the recommendation menu or enter the name of the device you wish to install in place of the pump.
2. Select the auxiliary output from the selection menu.

Note: If you have attached the sample changer (ST20A or ST20) this unit offers you an additional auxiliary output called "DISPENSER" (a relay make contact), see note in Section 1.4.2.

The auxiliary instrument with the modified parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

Menu tree



1.5.3 Add

Select this command when you wish to add an auxiliary instrument to the list. You can also add an auxiliary instrument directly to the list by pressing the <+> (plus) key. You are always shown the following mask:

Name
Auxiliary output Aux.3

1. Select the name from the recommendation menu or enter the name of the device you wish to install.

Note: If you install several auxiliary instruments with the same name, you must provide each one with a different flag to allow the titrator to distinguish between them, e.g. Pump/2.

2. Select the auxiliary output from the selection menu.

The auxiliary instrument with the appropriate parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

1.6 Auxiliary values

When you select this menu you are shown 20 auxiliary value memories: H1 - H20. You can assign results of a titration such as blank values and potentials to these memories using the **Auxiliary value** function. These are then entered automatically here together with the date (see Section 2.3.15).

Auxiliary value 1 = H1, Auxiliary value 2 = H2, etc..

You can also enter numeric values here as an auxiliary value and you can then call up these under different functions.

As auxiliary value H1 the ZnSO_4 solution with the concentration of 0.1 mol/L is stored that is used as volumetric solution for the titer determination of EDTA (see METTLER method M007, Section 10).

```
H1          c ( ZnSO4 )    0.1
H2          1.0
etc.
```

You can neither delete H1 to H20 nor add "H21" to the list. If you select H1, for instance, you are shown the following parameter mask:

```
Auxiliary value      H1
ID-text              c ( ZnSO4 )
Value                0.1
Date/time            00-00-0000  00:00
```

1. You can not change the name H1: The line will be skipped when the selector bar is moved.
2. Modify eventually the identification text.
3. Modify the value (do not modify it, if you use method M007!).
4. You can not enter the date. It refers to the determination of the auxiliary value using the method function *Auxiliary value* and is entered here together with the time automatically (see Section 2.3.15).

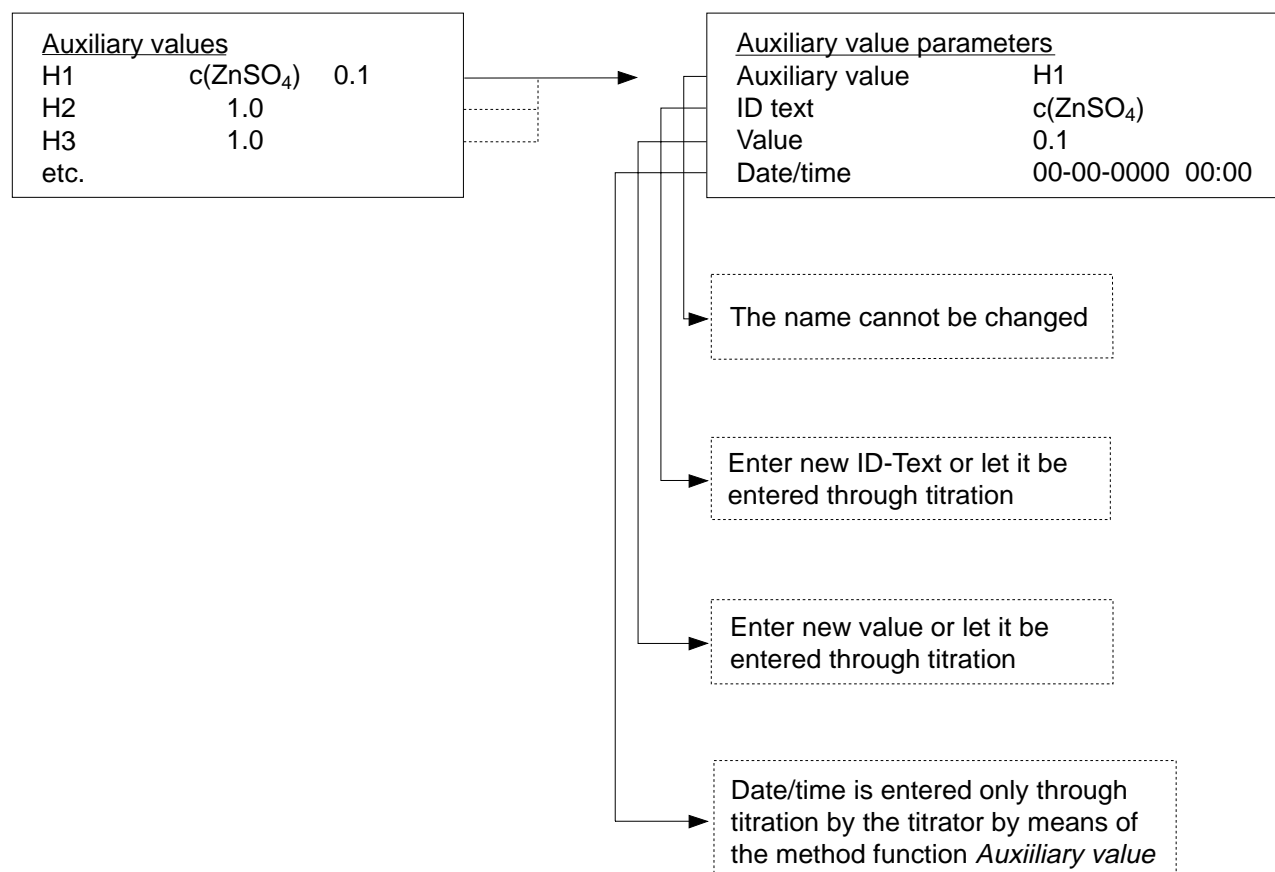
Notes: a. You can delete the data of an auxiliary value only by overwriting it or letting the titrator overwrite it through the *Auxiliary value* function.

b. You receive no warning from the titrator if you overwrite an auxiliary value. If you make full use of the auxiliary value memory you should print out a list of its values occasionally (see Section 5.1.2).

c. If you modify an auxiliary value parameter, the date/time is deleted.

The auxiliary value with the modified parameters is installed when you quit the parameter mask with **EXIT** (see the appropriate description at the end of Section 1.1.2).

Menu tree



1.7 Titration stands

When you select this menu you are shown the names of six possible titration stands with the specified parameters stirrer connection and default speed. The names of the titration stands are permanently installed, in other words you can not modify them.

Stand 1	Aux. 1	50
Stand 2	Aux. 1	50
ST20 1	Aux. 1	50
ST20 2	Aux. 1	50
Free stand	Aux. 1	50
Auto stand	Aux. 1	50

Stand 1 is the titration stand you receive with the standard equipment of the titrator. The associated stirrer is installed at auxiliary output Aux. 1.

Stand 2 is the second titration stand of a dual titration stand. The associated stirrer is installed at auxiliary output Aux. 1.

ST20 1 is the first sample changer titration stand. The associated stirrer is installed at auxiliary output Aux.1.

ST20 2 is the second sample changer titration stand. The associated stirrer is installed at auxiliary output Aux.1.

Free stand is a titration stand that you have set up independently of the titration stands mentioned, e.g. if you attach a stirrer to a stand. The associated stirrer is installed at auxiliary output Aux. 1.

Auto stand is the name of the titration stand at which you can run a sample series without ever being asked to insert the next sample (see Section 3.1.3). The associated stirrer is installed at auxiliary output Aux.1.
With this stand you can, for example, use a robot to change the samples.

DL70ES/DL67: "ST20 2" is not listed, as you can not connect a second sample changer.

DL77: When two sample changers are connected, one must be a **ST20A** (see Section 1.8.4).

You can neither delete a titration stand nor add a new one to the list. If you select Stand 1, for instance, you are shown the following parameter mask:

Name	Stand 1
Stirrer connection	Aux.1
Default speed [%]	50
Conditioning mode	Fix

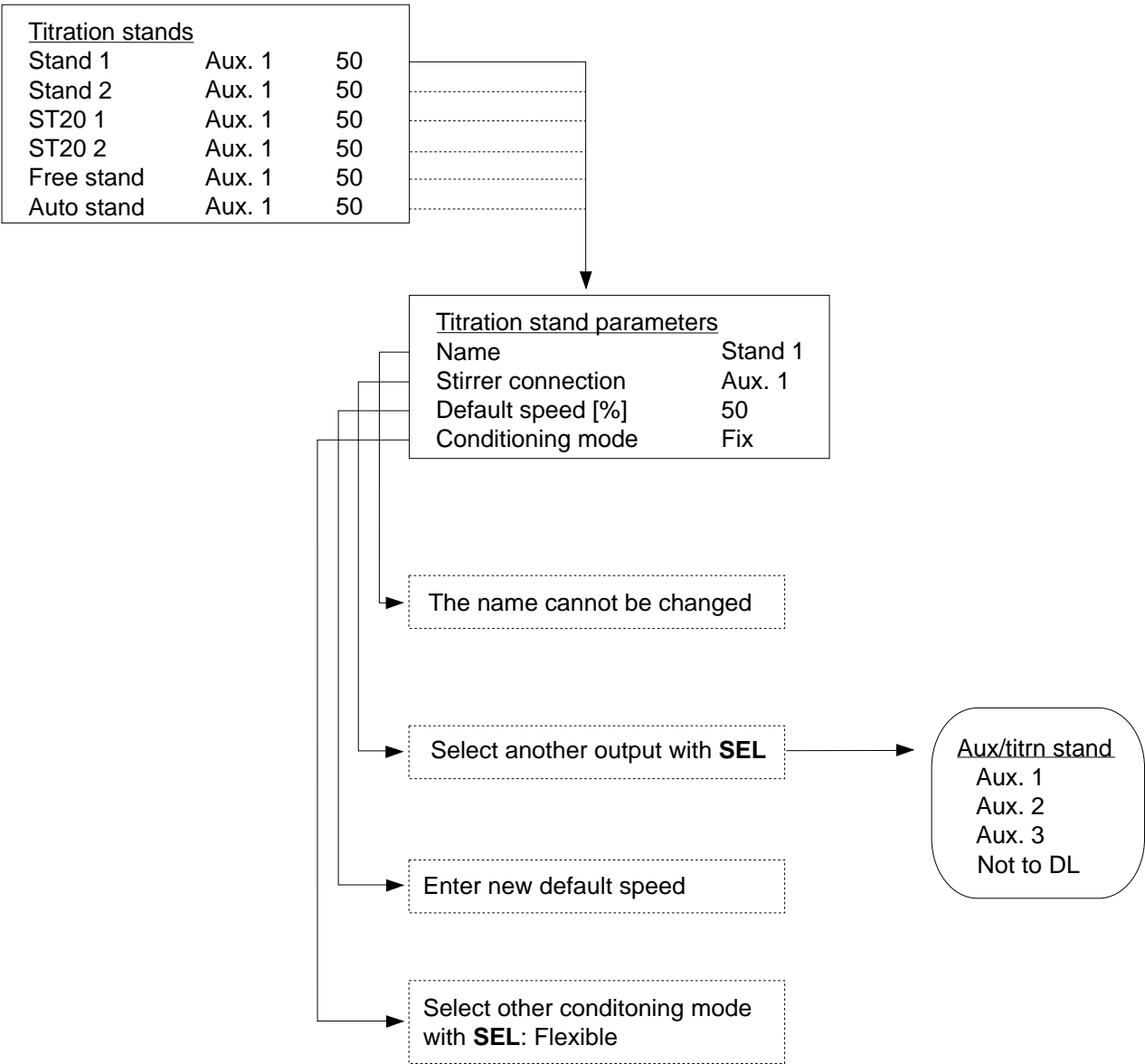
1. You can not change the name of the titration stand: The line is skipped when the selector bar is moved.
2. From the selection menu select the stirrer connection to which you wish to attach the stirrer for titration stand 1.

If you stir with a stirrer that is not attached to the titrator, select here **Not to DL**: The waiting times specified under the **Stir** function are always adhered to within the method. However, you yourself are responsible for the stirring (see *Stir* function, Section 2.3.3).

3. Enter the default speed [0-100%] of the stirrer:
 - 0 → the stirrer is inactive;
 - 100 → the stirrer operates at maximum speed.
4. Select the type of conditioning for the *Conditioning* function: "Fix" or "Flexible" (pertains only to stand ST20 1 or ST20 2).
 - Fix: The ST20A (ST20) conditions with the parameters designated in the *Conditioning* function (see Section 2.3.10).
 - Flexible: The ST20A (ST20) conditions every time it finds a conditioning beaker on the turntable.

Note: The titration stand in use is always recorded.

Menu tree



1.8 Peripherals

When you select this menu you are shown the names of the units you can attach to the titrator via the RS232C or CL interface. When you receive the titrator no unit is installed.

Printer	Not installed
Balance	Not installed
System	Not installed
Sample changer	Not installed

1.8.1 Printer

If you wish to attach a printer you have to install it. Select **Printer** and you are shown the following parameter mask:

Status	Not installed
Printer type	LX800
Paper	Fanfold
Paper format	8 ¹ / ₂ * 11"
Automatic form feed	No
Frame lines	Straight
Baud rate	2400
Parity	Even
Number data bits	8 bits
Number stop bits	1 stop bit

1. Select the status with **SEL**: "Not installed" or "Installed".
2. Select the printer type from the selection menu:
 - LX800 (EPSON ESC/P command language)
 - HP Deskjet (HP PCL III command language)
 - DICONIX 180si (extended IBM Proprinter command language)
 - IBM (IBM Proprinter command language)
 - Diabolo 630 (ASCII characters).
3. Select the paper with **SEL**: "Fanfold" or "Single sheet".
 - Single sheet: - A form feed is forced at the end of a page.
 - If you select **Yes** for "Automatic form feed" (see Parameter 5), a header and a footer will be printed on each page of the record.

Caution: Do not activate the printer's line feed or form feed functions if you have selected "Automatic form feed" for the "Single sheet" paper mode! On the **titrator** the following key combinations will activate the printer functions:

<index + L> causes a line feed,
<index + P> causes a form feed.

4. Select the paper format from the selection menu:
 - DIN A4 (width = 21 cm, length = 29,7 cm)
 - 8¹/₂ * 11" (width = 8¹/₂ inches, length = 11 inches)
 - 8¹/₂ * 12" (width = 8¹/₂ inches, length = 12 inches)
5. Select the automatic form feed with **SEL**: "Yes" or "No".
 - Yes: The printer inserts a form feed at the end of each document.
 - No: Each document is separated from the next by a space of 2 lines.
6. Select the frame lines for the record from the selection menu:
 - Straight: The printout will be framed with continuous lines.
 - Dotted: The printout will be framed with dashed lines; the printing proceeds at twice the previous rate.
 - None (no frame): Printing proceeds fastest with this parameter.
7. Select the baud rate from the selection menu:
 - 1200
 - 2400
 - 4800
 - 9600.
8. Select the parity from the selection menu:
 - Even
 - Odd
 - None.
9. Select the data bits with **SEL**: "8 bits" or "7 bits".
10. Select the stop bits with **SEL**: "1 stop bit" or "2 stop bits".

Attach the printer to the data output with the designation "Printer". The connection cable is part of the standard equipment of the titrator (see Section 11.3: *Accessories*).

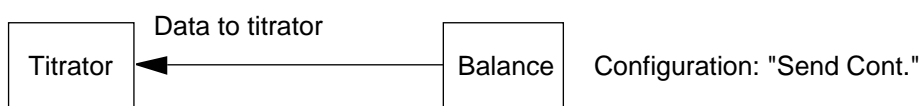
1.8.2 Balance

If you wish to attach a balance you have to install it. Select **Balance** and you are shown the following parameter:

Transmission mode Select from the selection menu

- Unidirectional
- Bidirectional
- Not installed

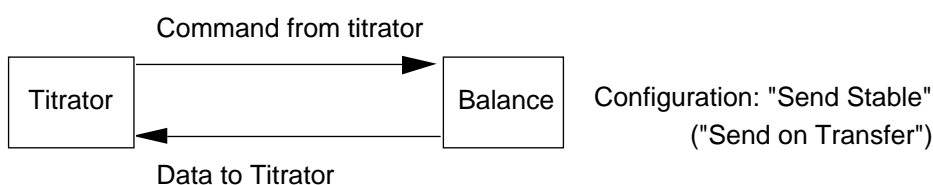
- **Unidirectional:** In the case of the weight request the balance transfers the current weight value (without stability detector) continuously and this is displayed by the titrator. You must confirm this display with **RUN** for the value to be accepted.



- **Bidirectional:** In the case of the weight request the balance transfers the current weight value when the titrator requests it and this value is then displayed by the titrator (with stability detector).

On AM, PM and AT balances the weight limits are superimposed on the balance display (see Section 3.1.1).

The titrator accepts the weight value when you press either the **RUN** or the transfer key of the balance.



Note: a. You can attach every METTLER balance fitted with a CL interface. The data output of the attached balance must be configured as follows:

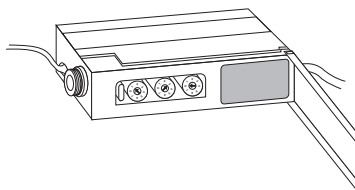
- Baud rate: 2400
- Parity: even
- Operating mode: "Send Cont." for unidirectional transmission mode
"Send Stable" ("Send on Transfer") for bidirectional transmission mode.

Notes: b. With AT balances, the following settings are important:

- | | | | |
|-------------|------|-------|----------------------------|
| • Unit: | Prt | on | print/transfer command on |
| | | g | balance unit in g |
| • Int-FACE: | SENd | S.Stb | transmission mode: standby |
| | bd | 2400 | baud rate: 2400 |
| | PAr | -E- | parity: even |
| | HS | OFF | handshake (XON/XOFF) off |

c. With AB, PB and PR balances, the LC-CL cable must be configured as follows:

- Left switch: position 7
- Middle switch: position 3
- Right switch: position 4



d. You can also attach SARTORIUS balances with the converter cable RS-CL/CL-RS (see *Accessories*, Section 11.3); for this you must select **Bidirectional** as the transmission mode. We have tested the following balances:

- BA 3100 P
- E 12000 S
- A 200 S
- MC1 LC 220 S
- MC1 RC 210 P

The data output of the attached balances must be configured as follows:

- Data output: ext. print command / irrespective of stability
- Baud rate: 2400
- Parity: even
- Stop bit: 1
- Weight unit: g

1.8.3 System

If you wish to attach a terminal or a computer you must first install these devices. Select **System** and you are shown the following parameters:

Instrument type	Not installed
Baud rate	4800
Parity	Even
Number data bits	8 bits
Number stop bits	1 stop bit

1. Select the instrument type from the selection menu:
 - Color terminal
 - Monochrome term.
 - Computer
 - Not installed.
2. Select the baud rate from the selection menu:
 - 1200
 - 2400
 - 4800
 - 9600.
3. Select the parity from the selection menu:
 - Even
 - Odd
 - None.
4. Select the number of data bits with **SEL**: "7 bits" or "8 bits".
5. Select the number of stop bits with **SEL**: "1 stop bit" or "2 stop bits".

Terminal

You can use a terminal of the type DEC VT340 or DEC VT241 as a color terminal. As a B/W terminal a DEC VT330 or DEC VT240 terminal can be used. The cable with order number 201507 can be used as connection cable (see Section 11.3: *Accessories*).

The terminal should be configured as follows:

- Baud rate: 4800 or 9600 baud
- Parity: even
- Number data bits: 8
- Number stop bits: 1

You will find additional information regarding configuration of the terminal and keyboard operation in Section 7.2.

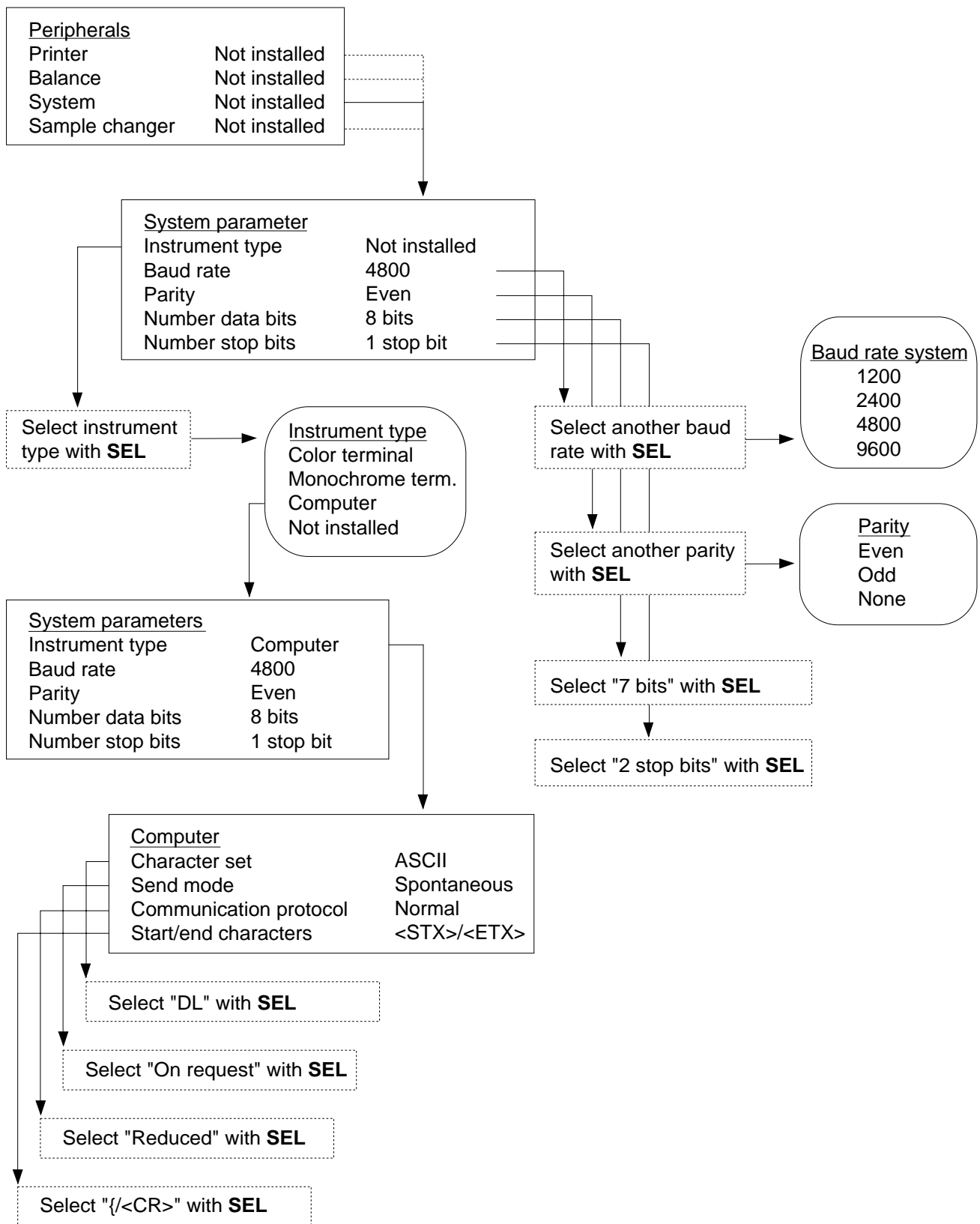
Computer

The computer requires an RS232C interface (DTE). The baud rate, parity, number of data bits and number of stop bits are freely selectable. The cables with order numbers 201507 (25 pin) or 201508 (9 pin) can be used as connection cable (see Section 11.3: *Accessories*).

If you have installed a computer, you must confirm the line "Instrument type ... Computer" with **RUN** to define the following parameters:

Character set	<p>Select the relevant parameter value with SEL: "ASCII" or "DL".</p> <p>ASCII: The standard character set (HEX 20 to HEX 7E) for text output to the computer is used.</p> <p>DL: The character set in the titrator is used.</p> <p>Caution: If you select DL, you must define 8 for the number of data bits!</p>
Send mode	<p>Select the relevant parameter value with SEL: "Spontaneous" or "On request".</p> <p>Spontaneous: The titrator sends the computer requests and data as soon as they are generated (assumes that the computer is ready).</p> <p>On request: The titrator awaits the appropriate inquiry from the computer before sending a request or data.</p>
Communication protocol	<p>Select the relevant parameter value with SEL: "Normal" or "Reduced".</p> <p>Normal: The data received either by the titrator or the computer will be checked and errors found will be announced with error messages (safety mechanism active).</p> <p>Reduced: Data received will neither be checked nor acknowledged, consequently no error messages will be sent (safety mechanism inactive).</p>
Start/end characters	<p>Select the relevant parameter value with SEL: "{/CR}" or "<STX>/<ETX>".</p> <p>These start of text and end of text characters for telegrams to be sent depend on the input possibilities of your computer.</p>

You will find additional information regarding communication between the titrator and the computer in Section 7.1 and in the Operating Instructions provided with the RS option.

Peripherals: Menu tree for system with computer

1.8.4 Sample changer

If you wish to attach the sample changer (ST20A or ST20) you have to install it. Select **Sample changer** and you are shown the following parameter:

Status Select the relevant parameter with **SEL**: "Not installed" or "Installed".

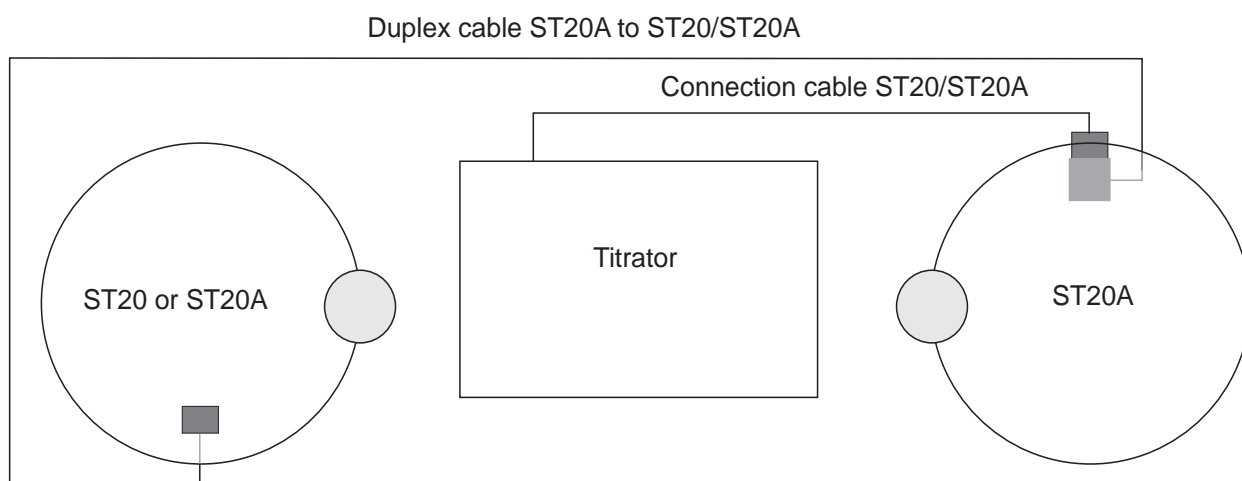
DL77: This titrator can control two sample changers (see Section 1.7: *Titration stands* "ST20 1" and "ST20 2"). The condition for this is that you must use at least one new sample changer, the **ST20A** (**ST20** is the name of the older model).

The second sample changer is connected to the first with the "duplex cable ST20A". You can designate the two sample changers ("ST20 1" and "ST20 2") using the identification switch of the ST20A (see the ST20A installation instructions).

Two sample changers can be used for

- **parallel** titrations of a sample series on each of the sample changers (Analysis A and Analysis B, see Section 3.14).
- **successive** determinations of sample series with a total of 40 samples ("unattended"). The conditions for this are described in Section 3.15.

Connection scheme for 2 sample changers



1.9 Miscellaneous

When you select this menu you are shown the various submenus and parameters whose values or names you can or must select or specify:

Date/time format
Enter date/time
Language
Record header
Titrator ID
Routine level
Audio signal
Analysis parameters

1.9.1 Date/time format

You are offered several ways to write the date and time. If you wish to modify the existing formats, select this parameter and you are shown the following parameters:

Date format	Select the new format from the selection menu.
Time format	Select the other format with SEL .

Note: If you modify the format after one month, for example, the new format will be adopted for all previously stored data.

1.9.2 Enter date/time

In the course of time you may possibly need to reset the dates. When you select this line you are shown the following parameters:

Day	Enter the appropriate data.
Month	
Year	
Hour	
Minute	

1.9.3 Language

The titrator understands and outputs English, German, French, Italian, and Spanish. Select this parameter if you wish to change the current language and you are shown the following parameter:

Active language Select the new language from the selection menu.

1.9.4 Record header

Select this parameter if you wish to enter a text that should appear on every record of a titration method. You are shown the following parameter mask:

Text : You have these two lines available for entry.
Text :

1.9.5 Titrator ID

Select this parameter if you wish to enter an identification for **your** titrator – it will appear in every record header. You are shown the following parameter:

Titration ID Enter an identification.

1.9.6 Routine level

With the aid of this menu you set up a precondition that determines whether all people who work with the titrator should also have access to the main menus, in other words be allowed to delete installation data or modify methods. If, for example, you have temporary staff who can perform only routine analyses, it is practical to "block" their access to several menus.

When the titrator is delivered you have access to all menus, they are "open". To change this situation, select **Routine level**. You are shown the following parameter mask:

Installation	"Open": The user has access to this menu. If he should not access it, press SEL : → "Blocked".
Editor	"Open": The user is allowed to develop, modify, delete, and print out methods in the Editor menu. If he should be allowed to only print out the stored methods press SEL : → "Blocked".
Documentation	"Open": The user has access to this menu. If he should not access it, press SEL : → "Blocked".
Analysis: Modify parameters	"Open": The user is allowed to modify the parameters of the current method in the Analysis menu. If he should not be allowed to do so, press SEL : → "Blocked".

When you now select **Routine** in the USER LEVEL menu the user no longer has access to the menus blocked here (see Section 6).

1.9.7 Audio signal

An audio signal either confirms each keystroke or draws your attention to instructions, directions or error messages. In the factory setting of the titrator, all these parameters are signalled. When you select this menu you are shown the following parameter mask:

Keystroke	"Yes": You hear a brief signal after every keystroke. If you do not wish to hear this, press SEL : → "No".
Results	"Yes": You hear a brief signal after every result that appears on the display. If you do not wish to hear this, press SEL : → "No".
Messages	"Yes": Your attention is drawn to error messages, directions and instructions by a signal. If you do not wish to hear this, press SEL : → "No".

- Notes:
- Error messages:** You must confirm each error message with **RUN** and rectify the error. Otherwise the titrator will not continue with the desired operation.
 - Instructions:** Your current method includes the function **Instruction** under which you have entered a text. You must confirm this with **RUN** before the titrator can continue operation (see Section 2.3.6).
 - Directions:** During the sequence of a method the titrator will give you directions that you must confirm with **RUN** before it can continue operation.

1.9.8 Analysis parameters

During the course of a titration method, certain information, which must be confirmed with **RUN** for the titration to proceed, will appear on the display:

- Initially the mask "Installed are" (see Section 3.1).
- After the titration the mask "Results of this sample" (see Section 3.1.3).

These two masks can be excluded to accelerate the titration course. If you select this menu, you are shown the following parameter mask:

Installation data	"Yes": The mask "Installed are" appears prior to titration of each sample. Should this not happen, press SEL : → "No".
Results last sample	The mask "Results of this sample" appears after titration of each sample. Should this not happen, press SEL : → "No".

Contents	Page
2. EDITOR	2-3
2.1 Select methods	2-5
2.1.1 Print	2-6
2.1.2 Delete	2-7
2.1.3 Modify	2-7
2.2 Select functions	2-8
2.2.1 Cut	2-8
2.2.2 Copy	2-8
2.2.3 Paste	2-8
2.2.4 Add	2-9
2.2.5 Modify	2-10
2.2.6 Save method	2-11
2.3 Functions	2-14
2.3.1 Title	2-14
2.3.2 Sample	2-15
2.3.3 Stir	2-18
2.3.4 Measure	2-20
2.3.5 Temperature	2-22
2.3.6 Instruction	2-23
2.3.7 Dispense	2-24
2.3.8 Pump	2-25
2.3.9 Rinse	2-26
2.3.10 Conditioning	2-27
2.3.11 Auxiliary instrument	2-31
2.3.12 Titration	2-32
2.3.12.1 DOS (Titration mode: dispensing)	2-34

2.3.12.2	EQP (Titration mode: equivalence point determination)	2-36
	Predispensing	2-39
	DYN (Titrant addition)	2-40
	INC ((Titrant addition)	2-41
	EQU (Measure mode)	2-42
	TFIX (Measure mode)	2-44
	Threshold (Equivalence point recognition)	2-45
	EQP range (Equivalence point recognition)	2-49
	Termination criteria	2-51
	Evaluation criteria	2-51
2.3.12.3	EP (Titration mode: end point determination)	2-53
	Predispensing	2-53
	Continuous (Titrant addition)	2-54
	Dynamic (Titrant addition)	2-55
	End point mode	2-56
	Tendency	2-57
	Maximum volume	2-57
2.3.12.4	LEARN EQP (Learn titration: equivalence point determination)	2-58
2.3.12.5	LEARN EP (Learn titration: end point determination)	2-60
2.3.13	pH/mV-stat	2-62
2.3.14	Calculation	2-66
2.3.15	Auxiliary value	2-70
2.3.16	Titer	2-71
2.3.17	Calibration	2-72
2.3.18	Statistics	2-75
2.3.19	Record	2-77
2.3.20	Sync	2-81

2. EDITOR

In this menu you develop **titration methods**, which you can store. You can modify or delete these methods or print them out.

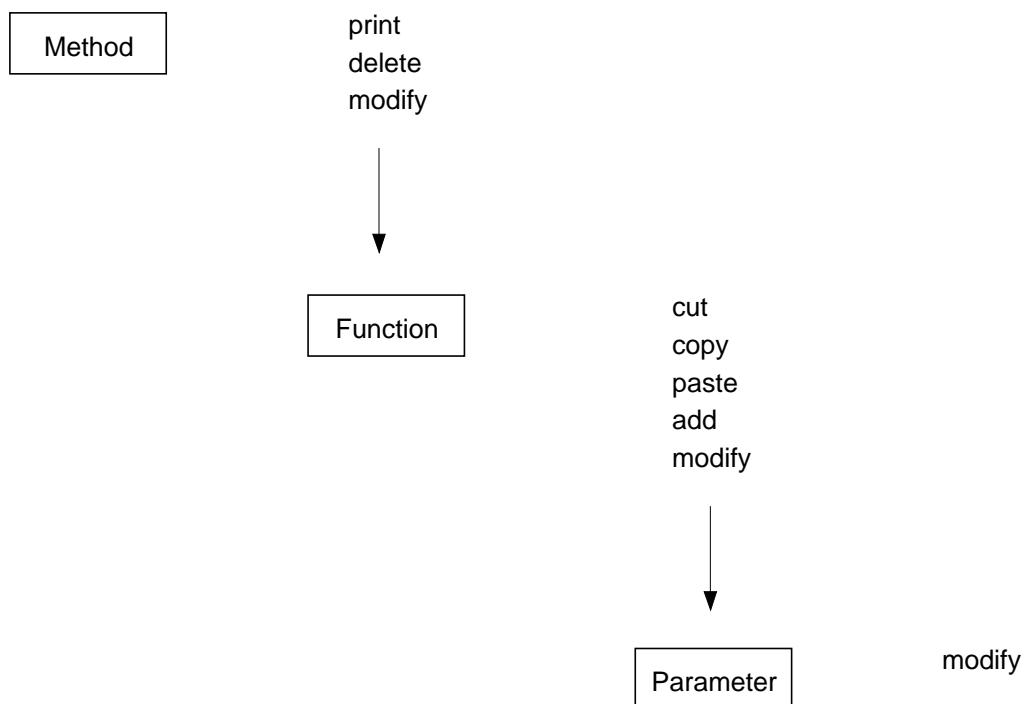
A complete titration method comprises sample preparation, dispensing of auxiliary solutions, stirring and waiting times, the titration itself, calculation of the result and statistics and a record. In the titrator these substages are defined as **functions** that are executed in **succession** in an analysis. Within a method you can not only modify these functions, but also cut them out of the defined sequence and hence delete them or paste them in at a different place. You can also add a new function to a method. Virtually all functions can occur more than once in a method (see Section 8.5.1).

List of all functions:

- Title
- Sample
- Stir
- Measure
- Temperature
- Instruction
- Dispense
- Pump
- Rinse
- Conditioning
- Auxiliary instrument
- Titration
- pH/mV-stat
- Calculation
- Auxiliary value
- Titer
- Calibration
- Statistics
- Record
- Sync

The individual functions comprise **parameters** whose values or names can be modified.

When you develop a new method you are always shown the **standard method** with default parameters, which you can then modify to suit your needs (see: *New Method*, Section 2.1).



You modify a method by changing its functions.

You modify a function by changing its parameters.

You will find the representation of the Editor menu tree at the end of Section 2.2.

2.1 Select methods

You are shown the following submenu in the Editor menu:

Method ID
METTLER methods
User methods
New method

Under method ID you can call up all stored methods,
under METTLER methods only those in the application data base,
and under user methods only those methods available in the user data memory.

Method ID

The method identification is a 4-character, alphanumeric identifier of a method. If you know it, select **Method ID** (see *Title* function, Section 2.3.1).

Enter the method ID and confirm with **RUN**. The entry will be masked by the following selection menu:

Print
(Delete) appears only after ID entry of a user method
Modify

Note: It is possible to enter an asterisk (*) or a question mark (?) **after** the first, second or third letters/numbers of the method ID:

- *: replaces all subsequent letters/numbers. For example, x*: results in the display of all methods with an ID beginning with this letter or number.
- ?: replaces one letter or number within the ID. For example, xy?z will result in the display of all methods with an ID containing these three letters/numbers.

METTLER methods

When you select this option you receive a list of all methods stored in the application data base with the identification and the name of the method:

M001 Acid content
M002 Titer of NaOH (0.1 mol/L)
etc.

If you select M001, for instance, the method is masked on the right by the following selection menu:

Print
Modify

User methods

If you select this, you are shown a list of methods with the identification and name of the method if you yourself have stored a method in the user data memory, e.g.

KM59 pH-stat of Gerusil

UB33 H₃PO₄ in cola drinks

If you then select a method, it is masked on the right by the following selection menu:

Print

Delete

Modify

New Method

If you wish to develop a new method, select **New Method**. You are shown the standard method with the following functions:

Title

Sample

Stir

Titration

Calculation

Statistics

Record

All parameters of the functions – except those of the **Title** function – are defined with default values or names. You can accept or modify these; you can also add additional functions to this standard method (see Sections 2.2.3 and 2.2.4).

Note: To set up a method with more than the standard functions, you will find the scheme in Section 8.8 useful. You can copy this scheme and use it to design the method, which you can then enter in this menu.

2.1.1 Print

– Position the selector bar on this command and confirm with **RUN**.

The method together with its functions and the corresponding parameter values will be printed out on an attached printer.

2.1.2 Delete

- Position the selector bar on this command and confirm with **RUN**.

In the selection menu "Delete method" you can confirm or cancel the selection:

- Yes: The method is deleted.
- No: The method remains stored.

Notes: a. METTLER methods can **not** be deleted.

- b. If a user method is entered in the method list of the Analysis menu and not yet executed, the message "Method is blocked" appears (see also Note c. in the next section).

2.1.3 Modify

When you select this command you receive a list with all functions included in this titration method, e.g.

Title
Sample
Measure
Dispense
Stir
Titration
Calculation
Record

Notes: a. You can modify METTLER methods to match your requirements, but you must then provide them with a new method identification under the **Title** function in order to save them (see Section 2.3.1).

- b. If you modify a user method or simply wish to **copy** it and keep the original, you must give it a new method identification under the **Title** function (see Section 2.3.1).
- c. If you select a user method that is entered in the method list of the Analysis menu and not yet executed, the message "Method is blocked" appears. To modify this method, you have to give it a new method identification under the **Title** function. The method in the method list remains unchanged (see Sections 3.1 and 3.13).

2.2 Select functions

When you select the **Title** function, you are shown this selection menu on the right of the display:

Modify

You can only modify the **Title** function since the storage and call options require that it is always first in the sequence of functions.

If you select the **Measure** function you are shown the following selection menu on the right of the display.

Cut

Copy

Paste

Add

Modify

2.2.1 Cut

- Position the selector bar on the command and confirm with **RUN**. (You can also cut a function directly from the list by pressing the <-> (minus) key.)

The **Measure** function is **deleted** at this location.

However, it is stored in a buffer memory so that you can paste it in elsewhere if appropriate (see Section 2.2.3).

The function remains stored until you either cut or modify another function.

2.2.2 Copy

If you merely wish to copy the **Measure** function in order to paste it in elsewhere,

- position the selector bar on this command and confirm with **RUN**.

The **Measure** function remains at this location and is also stored in a buffer memory so that it can be pasted in elsewhere.

The function remains stored until you either cut or modify another function.

2.2.3 Paste

You now wish to paste in the cut (or copied) **Measure** function **before** the **Stir** function:

- Position the selector bar on the **Stir** function and press the <=> (equals) key.

The **Measure** function now follows the **Dispense** function:

Title
Sample
Dispense
Measure
Stir
Titration
Calculation
Record

You can paste a function only if you have first cut or copied it. Since the **Measure** function is still stored, you can also paste it in ahead of the **Titration** function, for instance.

2.2.4 Add

You would like to add the **Auxiliary value** function to the method, in this case **before** the **Record** function:

- Position the selector bar on the **Record** function and press the <+> (plus) key. You are now shown a list of all functions (except *Title* function).
- Position the selector bar on the **Auxiliary value** function and confirm with **RUN**.

The **Auxiliary value** function now follows the **Calculation** function:

Title
Sample
Dispense
Measure
Stir
Titration
Calculation
Auxiliary value
Record

Note: If you select an improper sequence when pasting or adding the functions, the titrator sends you an error message (e.g. you can not place the **Statistics** function before the **Calculation** function). However, the error message is not outputted until the method is saved.

2.2.5 Modify

This command shows you a mask with the **parameters** needed for the function. You can modify the values or names of these parameters.

Notes: a. The parameters of all functions – except those of the **Title** function – are defined by default values or names. These are overwritten as soon as you enter a new value or name. If you wish to correct a parameter, indicate the appropriate letter (number) with the cursor (with → or ←), then enter the new one. With **SEL**, other parameters may be selected from the recommendation or selection menus (see Section 1.1.2).

b. The method identification, formulae and conditions are checked immediately after their entry. If they are incorrect, an error message appears, such as:

```
Error No.3
Wrong formula
Modify
Terminate
```

If you confirm "Modify" with **RUN**, you can change the entry.

If you confirm "Terminate" with **RUN**, the most recently saved prior entry appears.

c. As soon as you quit a parameter mask with **EXIT** the values or names are stored. The selector bar must be located on a parameter here. If you confirm the title line of a parameter mask with **EXIT**, the following mask appears:

```
Save modifications?
      Yes
      No
```

If you confirm "Yes" with **RUN**, the titrator stores the changed values or names.

If you confirm "No" with **RUN**, the old remain stored.

The display then shows either the method functions or the master parameter mask.

d. If you press a **key combination** (<index + letter>) to quit the Editor menu, the following selection menu appears:

```
Save?
      Yes
      No
```

If you confirm "Yes" with **RUN**, the titrator stores the changed method.

If you confirm "No" with **RUN**, the old values remain stored.

e. If you do not wish to modify the parameters of a function, after you have checked them, quit the mask with **Exit**. The location of the selector bar is immaterial here. The values of this function remain stored.

2.2.6 Save method

After modification of each function you can save your method.

- Press **EXIT**, regardless of the current position of the selector bar. The selection menu "Save"? appears:

No: Confirm with **RUN**: The method with the modified parameters is not saved, the old version is retained.

Yes: Confirm with **RUN**: The titrator now checks the method and, if several errors are present, draws your attention to the first one.

- Confirm these messages with **RUN** in each case.

a. You are shown the list of the method functions with the selector bar on the first incorrect function. After you have corrected its parameters and saved the method once more (see above) you are shown the next incorrect function, etc.

b. If you have entered a method identification under the **Title** function that already exists, the selection menu "ID exists" appears:

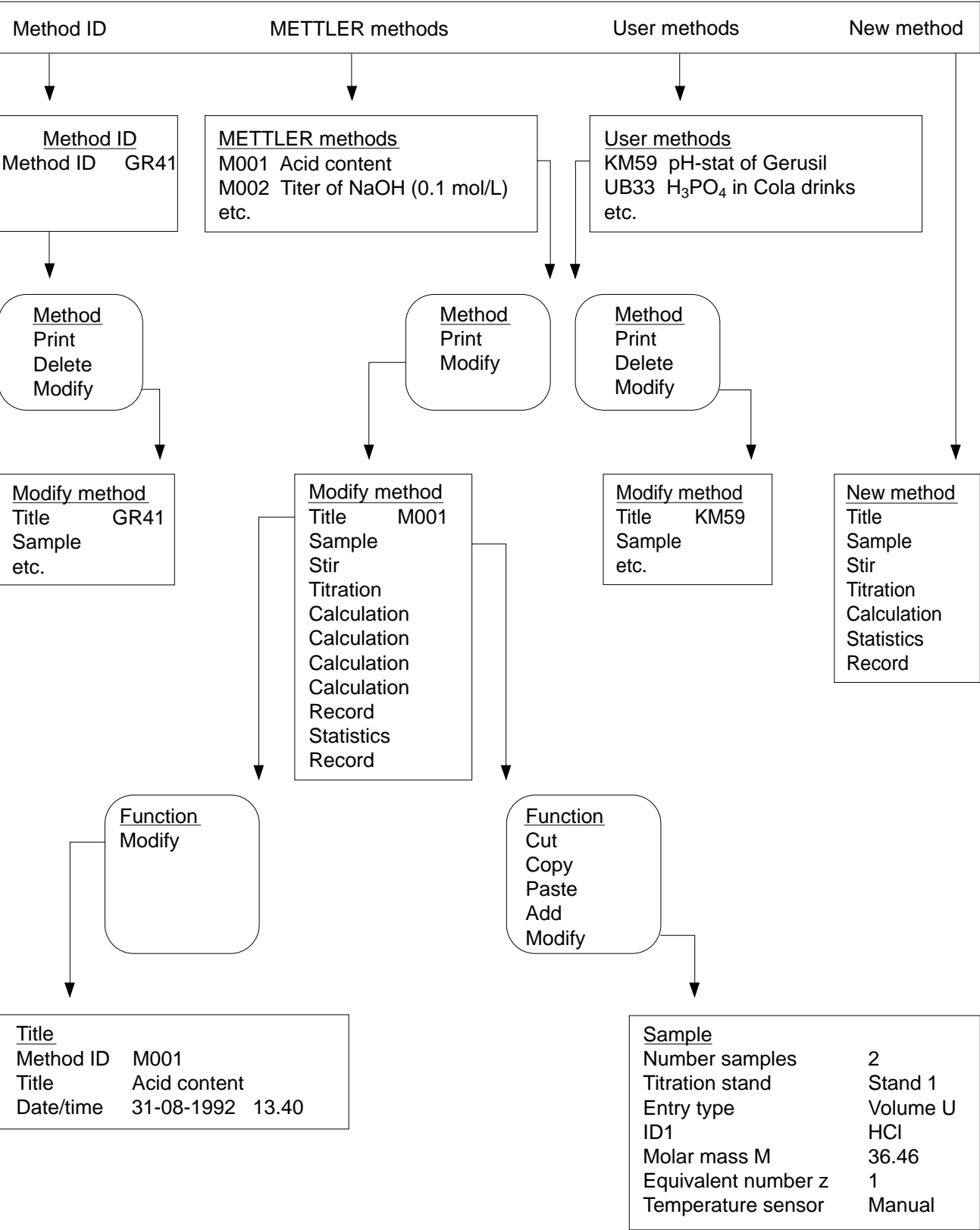
Overwrite ID: Confirm with **RUN**: The new or modified method is saved, that with the same identification is deleted.

Modify ID: Confirm with **RUN**: You are shown a list of the method functions and can modify the method ID using the **Title** function. Then you can save the method (see above).

If you are not shown any error message when you confirm "Save?" with **Yes**, the list of user methods with the stored method appears in the display.

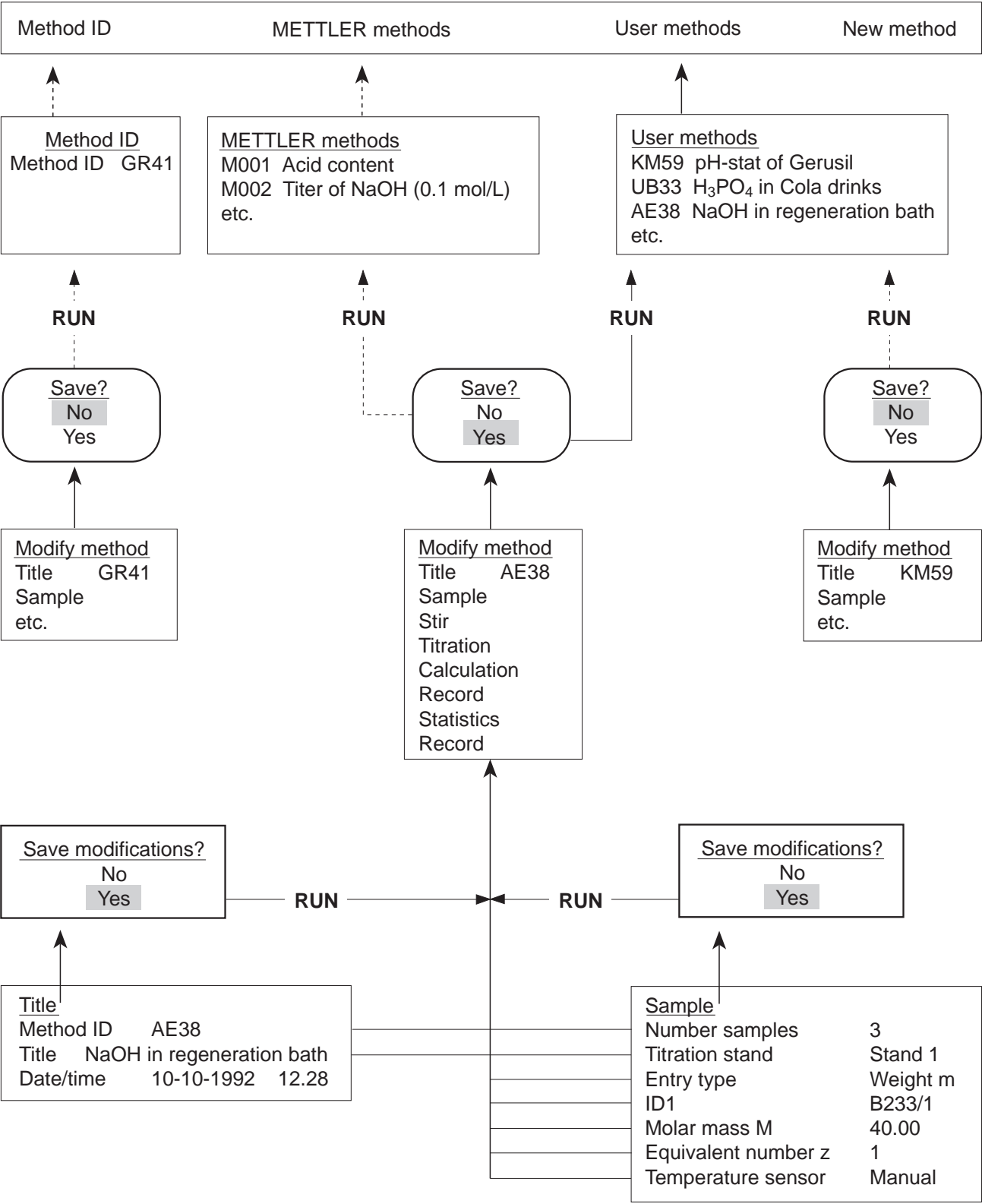
Note: If you press a key combination (<index + letter>) in the function list to quit the Editor menu, the selection menu "Save?" always appears first.

EDITOR: Menu tree



EDITOR: Menu tree – the way back

- The method/function is saved
- - - The method is not saved



2.3 Functions

All functions whose parameters you wish to modify have to be selected by the operation described under Sections 2.1 and 2.2. The following explanations of all functions and their parameters thus "take place" under the command "**Modify function**" (see above representation of menu tree).

2.3.1 Title

This function serves to identify the titration method. It must always be first in a method and is the only function that can occur only once in a method. You need to define its parameters. You call up the stored method in question using the method identification. The function has no significance for the progress of the titration.

1. Enter the method ID: letters and/or number, max. 4 characters, e.g. B101.
2. Enter a title for the method: e.g. "NaCl in butter".
3. Date and time are entered here automatically when you save a new or modified method. You can neither clear nor overwrite this information.

Notes:

- a. You must adhere to the upper case or lower case notation of the identification during entries, otherwise the titrator outputs an error message.
- b. You can **not** use any method identifications that start with upper case **M** as they are reserved for METTLER methods.
- c. Asterisks (*) and question marks (?) are not allowed in the method identification!

2.3.2 Sample

With the aid of this function you determine the parameters that are needed for the entry of the sample data such as weight or volume, and whether the temperature of the sample should be measured.

Parameters of the mask:

Number samples
Titration stand
Entry type
ID1
Molar mass M
Equivalent number z
Temperature sensor

1. Enter the number of samples **n**.

You can change **n** before and after the start of the method (see Section 3.1). A parameter value of $n = 3$, for instance, tells you that you have to titrate at least three samples, e.g. in a titer determination.

2. Select the titration stand at which you wish to determine the samples from the selection menu (see Section 1.7).

You can change the titration stand before the start of the method (see Section 3.1).

3. Select the type of entry from the selection menu:

a. Weight m

- Enter the lower weight limit [g].
- Enter the upper weight limit [g].

b. Volume U

- Enter the lower volume limit [mL].
- Enter the upper volume limit [mL].

The actual weight or volume are not entered until the samples are prepared or the titrator requests the amount after the start of the method (see Section 3.1).

If you violate this specified upper or lower limit you will be given a notice.

c. Fixed volume U

- Enter the volume [mL].

You can not change this entry before the start of the method!

4. Enter an initial identification: ID1 (number or name for all samples of this **Sample** function).
5. Enter the molar mass M.
The auxiliary value "Hj" can be entered instead of the number, provided that the molar mass has been stored here (see Section 1.6).
You can change the molar mass M before the start of the method (see Section 3.1).
6. Enter the equivalent number z.
For **one Sample** function you can enter only **one** molar mass M and **one** equivalent number z for your calculation. If your sample contains several substances that have to be determined in the titration through equivalence points, you must enter their molar mass and equivalent number in constant C under the **Calculation** function (see Section 2.3.14).
7. Select the temperature sensor from the selection menu if you have attached one (see Section 1.3); if not, select "Manual". The temperature entered before starting the method will then be used during the run (see Section 3.1).
With the aid of this parameter, the temperature of the sample solution is automatically determined or acquired, respectively, before the start of the functions **Measure**, **Titration** and **pH/mV-stat**. The slope of the pH electrode is then corrected for the temperature in the pH value calculation (see Section 1.3: *Temperature sensors* and Section 4.7: *Calibration of the temperature sensors*).

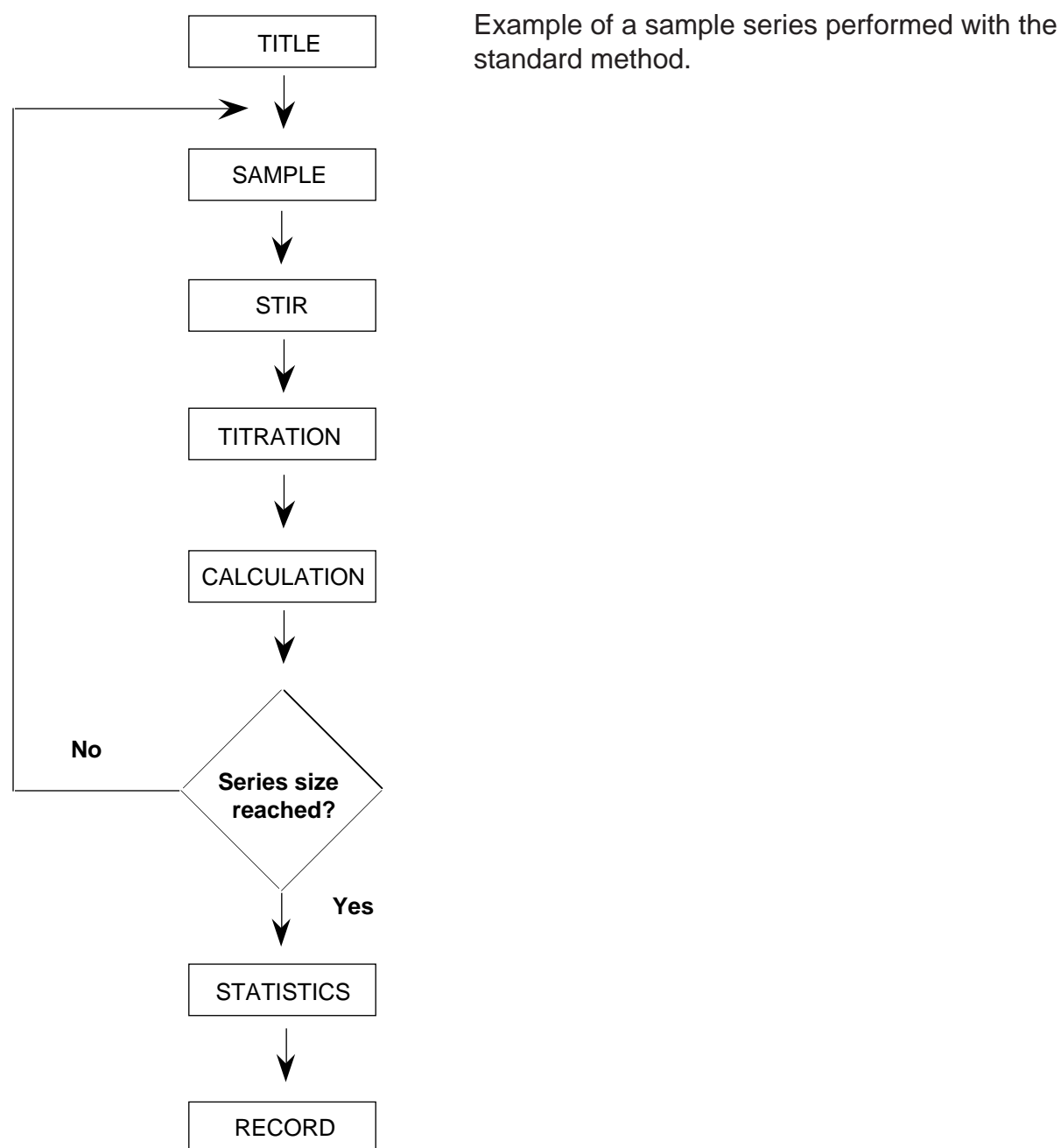
Important

The **Sample** function fulfills two additional tasks within the method sequence:

1. It provides the titrator with information regarding which **titration stand** is used for titration. The titration stand is linked with the stirrer connection and the default speed (see Section 1.7).
As soon as the titrator has executed the **Sample** function, it starts to stir at the default speed.

If there is no **Sample** function in a method, the titrator does not stir as it has no information regarding the titration stand. (A method with the **Stir** function but without the **Sample** function can not be saved for this reason!)
In this case you can enter the command to stir via the auxiliary function **Stirrer** (see Section 4.2).
2. For the titrator the function is the identifier for the start of a series (**n > 1**), in other words all functions that lie between the **Title** function and the **Sample** function will not be executed after titration of the first sample.

The identifier for the end of a series is the **Statistics** function. The titrator repeats all functions within the **loop** Sample/Statistics n times. If the **Statistics** function is missing in your method, only the first sample will be titrated, i.e. n is set to 1.



Note: You will find examples of methods in Section 8.7.

2.3.3 Stir

With the aid of this function you determine the stirring or waiting times and specify new stirring speeds.

Caution: It is essential that the **Stir** function is preceded by a **Sample** function within a method (see *Sample* function, page 16: *Important*).

1. Enter the speed of the stirrer [%]:
 - 0 → stirrer is inactive;
 - 100 → stirrer operates at maximum speed.
2. Enter the time [s]:
 - "0" means that the titrator will not wait at all,
 - "10" that it will wait 10 s before it starts the next function: It then stirs at the specified speed in each case. The running stir time is displayed.
3. Select a condition:
 - No.
 - Yes: Enter the condition.

Notes: a. You can set a condition for most of the functions. If this is met, the function is executed; if it is not met the function will be skipped (see *Functions with a condition*, Section 8.3 and an additional example under the *Record* function, Section 2.3.19). If you have not selected a condition the function is executed.

b. The speed you have specified applies to all following functions up to the next **Sample** or **Stir** function.

c. However, during a titration you can change the specified speed (see Section 3.10).

Example A: If you would like to continue stirring for some time after sample addition to dissolve a solid before the titrator starts the next function, you must add the **Stir** function after the **Sample** function.

Speed = e.g.: 60

Time = e.g.: 30.

The titrator stirs for 30 s at 60% maximum speed before it processes the next function. It continues to stir at this speed until the next **Sample** or **Stir** function.

Example B: If you would like to wait after the sample addition before starting stirring and before the titrator starts the next function, set

Speed = 0

Time = e.g. 30.

The titrator will not stir for 30 s before processing the next function. This must be a second **Stir** function to ensure that the titrator starts to stir:

Speed = e.g.: 70

Time = 0.

The titrator stirs at 70% maximum speed and immediately begins to process the next function (see example in Section 8.7.1).

2.3.4 Measure

You can use this function to measure the potential of a solution under defined conditions. The titrator acquires the measured value as raw result E (see notes at the end of this section). If you have selected a temperature sensor in the **Sample** function, the temperature of the sample solution is measured automatically before the titrator executes this function. If no sensor is attached (parameter "Manual"), the titrator adopts the temperature entered at the start of the method. The slope of the pH electrode is then corrected for the temperature in the pH value calculation (see Section 4.7: *Calibration of the temperature sensors*).

Caution: Stirring is performed under this function only if preceded by the **Sample** function (see *Sample* function, page 16: *Important*).

Parameters of the mask:	Sensor
	Unit of meas.(urement)
	ΔE [mV]
	Δt [s]
	t(min) mode
	t(max) [s]
	Condition

1. Select the sensor from the recommendation menu or enter one you have installed (see Section 1.2).
2. Select the unit of measurement: "mV" or "As installed".
"As installed" refers to the unit of measurement you have specified for the sensor (see Section 1.2.2).
3. Enter ΔE [mV]: e.g. 1.
4. Enter Δt [s]: e.g. 2.

Note: The drift in the electrode potential must be less than $\Delta E/\Delta t$ (0.5 mV/s) during the period Δt (2 s) if the potential is to be acquired as a measured value. This occurs within a defined time interval t(min) and t(max).
(See representation in Section 2.3.12.2: *Titration mode EQP, Measure mode EQU*).

5. Select t(min) from the following menu:

a. Fix: Enter t(min) [s]: e.g. 3.

Instead of a fixed time you can select a condition: The measured value must be greater or less than a specified set value.

b. $E >$ (greater than) set value: Enter set value [mV, pH, ...].

c. $E <$ (less than) set value: Enter set value [mV, pH, ...].

6. Enter t(max) [s]: e.g. 30.

Note: If you select 5b (5c) the titrator will start the next function only when the measured potential E is greater (less) than the set value and the drift condition is satisfied, but at the latest after t(max).

7. Select a condition:

– No.

– Yes: Enter condition.

Notes: a. The *Measure*, *Temperature*, *Dispense*, *Titration* and *pH-stat* functions generate **raw results** (see *List of designations*, Section 8.1). You can

- print these out as such on an attached printer (see Section 2.3.19 and Section 8.1.1 for exceptions).
- incorporate them in the calculation (see *Examples of formulae*, Section 8.6.1).
- save as a result if you have assigned them to the result **R**, e.g. $R = E$ (see Section 2.3.14).

b. At the end of a titration only the final results are shown on the display of the titrator, the raw results can only be printed out.

c. The titrator stores raw results up to the titration of the next sample within a loop (see Section 8.5.7).

2.3.5 Temperature

You can use this function to measure the temperature of a solution under defined conditions. The titrator acquires the measured value as raw result T (see Note a. in Section 2.3.4).

Parameters of the mask:

Sensor
Unit of meas. (urement)
ΔT [$^{\circ}\text{C}$, $^{\circ}\text{F}$, K]
Δt [s]
t(min) mode
t(max) [s]
Condition

1. Select the temperature sensor from the selection menu (see Section 1.3).
2. Select the unit of measurement from the selection menu: " $^{\circ}\text{C}$ ", " $^{\circ}\text{F}$ " or "K".
3. Enter ΔT [$^{\circ}\text{C}$, $^{\circ}\text{F}$, K]: e.g. 0.02.
4. Enter Δt [s]: e.g. 2.

Note: The temperature drift must be less than $\Delta T/\Delta t$ (0.01 $^{\circ}\text{C}/\text{s}$) during the period Δt (2 s) if the temperature is to be acquired as a measured value. This occurs within a defined time interval t(min) and t(max).

5. Select t(min) from the following menu:

- a. Fix: Enter t(min) [s]: e.g. 3.

Instead of a fixed time you can select a condition: The measured value must be greater or less than a specified set value.

- b. $T >$ (greater than) set value: Enter set value [$^{\circ}\text{C}$, $^{\circ}\text{F}$, K].
- c. $T <$ (less than) set value: Enter set value [$^{\circ}\text{C}$, $^{\circ}\text{F}$, K].

6. Enter t(max) [s]: e.g. 30.

Note: If you select 5b (5c) the titrator will start the next function only when the measured temperature is greater (less) than the set value and the drift condition is satisfied, but at the latest after t(max).

7. Select a condition:
 - No.
 - Yes: Enter condition.

2.3.6 Instruction

This function allows you to intervene manually in the sequence of the titration method, in other words the method is interrupted. The entered text appears at the desired position during the current method in the display and is supported audibly. The method does not continue until you confirm the instruction with **RUN**.

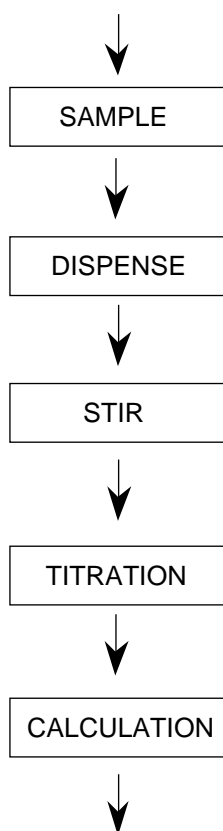
1. Enter the instruction.
2. Select a condition:
 - No.
 - Yes: Enter the condition.

2.3.7 Dispense

With the aid of this function you dispense a specified volume of titrant with a METTLER burette. The titrator acquires the dispensed volume as the raw result VDISP [mL] or QDISP [mmol] (see *List of designations*, Section 8.1 and the *Measure* function, Section 2.3.4).

1. Select the titrant from the recommendation menu or enter one that you have installed (see Section 1.1).
2. Enter its concentration [mol/L].
3. Enter the volume [mL].
A formula, such as "R1 + 0.5", can be entered instead of a number (R1 is the result of a **Calculation** function preceding this function).
4. Select a condition:
 - No.
 - Yes: Enter the condition.

Example: For a simple **back titration**, you can add, e.g. the **Dispense** function to the standard method:



Sample

You have added your sample to the titration vessel and started the titration. The titrator immediately starts to stir.

Dispense

The titrator dispenses the amount of the selected titrant and acquires the raw result QDISP.

Stir

You have set a long stirring time to allow the sample to react with the titrant before the titrator starts the back titration.

Titration

The titrator starts the back titration with a second titrant and acquires the raw result Q (consumption of the titrant in mmol up to the equivalence point or endpoint).

Calculation

The formula for the content of the sample:

$R = (QDISP - Q) * C/m$ (see Section 8.6: *Examples of formulae*).

2.3.8 Pump

With the aid of this function you select a time-controlled pump at an auxiliary output in order to pump a specified volume of an auxiliary reagent into the titration vessel.

1. Select the auxiliary reagent from the recommendation menu or enter one you have installed (see Section 1.3)
2. Enter the volume [mL].
3. Select a condition:
 - No.
 - Yes: Enter the condition.

Notes: a. You have defined the pump speed [mL/min], which you need to determine experimentally for each auxiliary reagent, in the Installation menu by means of the dispensing rate (see Section 1.4.2).

- b. If you attach the pump tubing to the wrong connectors, you will evacuate instead of dispensing.

2.3.9 Rinse

You use this function only in connection with the sample changer (ST20A or ST20). This has a built-in rinsing unit that controls the attached pump for rinsing (output "RINSE" or "DOSE"). The tips of the sensors and burettes are rinsed in the middle beaker position (see *ST20* or *ST20A Operating Instructions*).

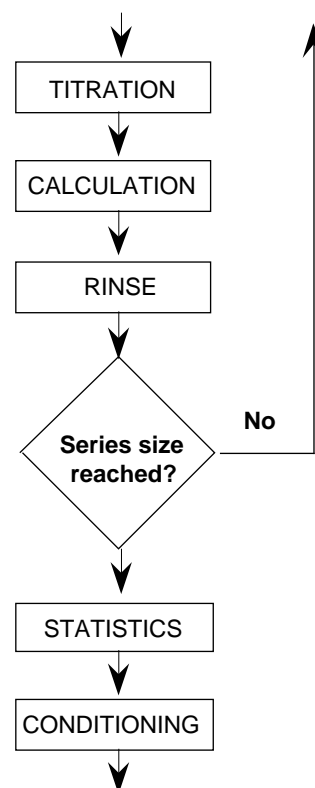
1. Select the auxiliary reagent from the recommendation menu or enter one you have installed (see Section 1.4).
2. Enter the volume [mL].
3. Select a condition:
 - No.
 - Yes: Enter the condition.

Notes: a. You have defined the rate of the rinsing process [mL/min], which you need to determine experimentally, in the Installation menu by means of the dispensing rate parameter (see Section 1.4.2).

- b. If you perform a **method series** on the sample changer and the titrator has titrated the **last sample** of the **last method**, sensor and burette tip are rinsed in the middle beaker position. The sample beaker is then raised so that the sensor does not dry out, in other words it remains in the sample solution.

To **prevent** this, you must add a **Conditioning** function to the last method and position a conditioning beaker after the final sample beaker.

The **Conditioning** function must then be **after** the loop of the last method so that the titrator does not perform conditioning after every sample determination (see Section 2.3.2: *Example of a sample series performed with the standard method* and Section 2.3.10: *Conditioning*).



2.3.10 Conditioning

You use this function only in connection with the sample changer (ST20A or ST20). You determine how often and how long a sensor is kept in a solution with stirring between individual samples or series to clean it or ensure its operational reliability.

1. Enter the interval (number of samples): e.g. 3.
2. Enter the time [s]: e.g. 60 → After three titrated samples conditioning is performed for 60 s. The running conditioning time is displayed.
3. Rinse: Should electrodes and burette tips be rinsed after conditioning?
 - No.
 - Yes: Select the auxiliary reagent from the recommendation menu or enter one you have installed (see Section 1.3).
Enter the volume [mL].

Caution: If you select this parameter the beaker can overflow after some time if you have inserted only one conditioning beaker in the turntable but condition the sensor after every sample.

4. Select a condition:
 - No.
 - Yes: Enter the condition.

Notes

When installing the sample changer titration stand you can select **Fix** or **Flexible** as conditioning mode (see Section 1.7).

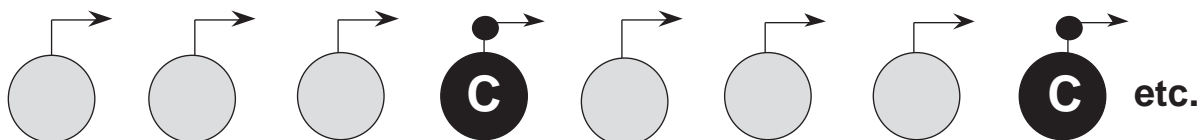
Fix: The ST20A (ST20) conditions with the parameters specified in this function (see below: Note 2).

Flexible: The ST20A (ST20) conditions whenever it finds a conditioning beaker (see below: Note 3). When titrating, in non-aqueous media for example, this mode allows you to place additional conditioning beakers where ever necessary **during** the method run.

1. Conditioning samples are marked on the sample turntable by red plugs.

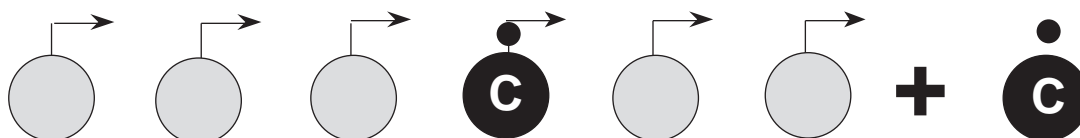
2. You have selected **Fix** as conditioning mode:

- a. You can follow the above example and insert and mark a beaker with the conditioning solution after every **third** sample:



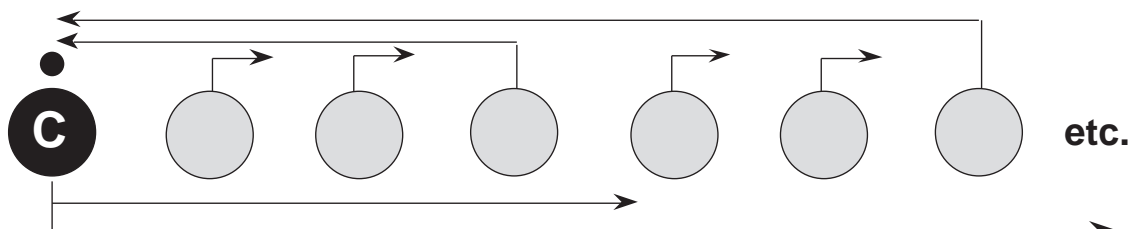
If you wish to condition the sensor on completion of a sample series, you must ensure that the number of samples can be divided by the interval number, e.g. samples = 9, interval = 3.

If, e.g. you have entered interval = 3 with 5 samples, the sensor and burette tips remain in the fifth sample solution. To prevent this, you must add an additional **Conditioning** function **after** the loop and insert a conditioning beaker after the last sample beaker (see Section 2.3.2: *Example of a sample series performed with the standard method*).

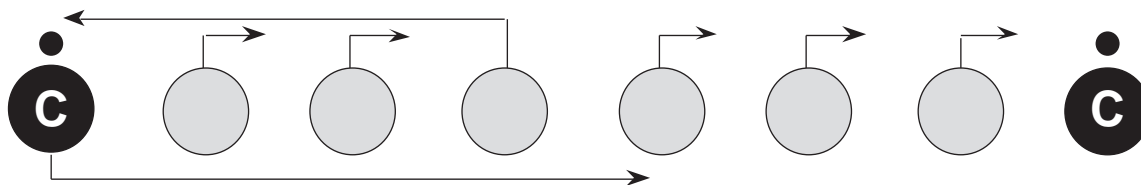


- b. You can insert and mark only one beaker with conditioning solution. If this does not directly follow the third sample, the ST20A searches for this conditioning beaker by rotating backward. It then conditions the sensor for 60 s. Afterwards, the sample turntable again rotates forwards to the fourth sample.

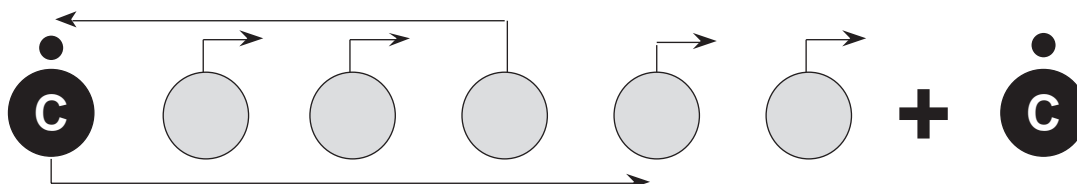
Caution: There must be no empty positions between the titration vessel of the last titrated sample and the conditioning beaker!



If you wish to condition the sensor on completion of a sample series, **always** place a conditioning beaker after the last sample beaker (number of samples must be divisible by the interval number!): The ST20A sees the following conditioning beaker:



If, e.g. you have entered interval = 3 with 5 samples, you must add an additional **Conditioning** function **after** the loop and place a conditioning beaker after the last sample beaker:



- c. You have entered **4** for the interval but have erroneously placed a conditioning beaker after the third sample beaker. The ST20A omits the conditioning beaker!

3. You have selected **Flexible** as conditioning mode:

- The method has no **Conditioning** function: The ST20A recognizes a conditioning beaker and conditions the sensor for 10 seconds.
- The method has a **Conditioning** function with parameters such as Interval = 4, Time = 30 s, Rinse volume = 5 mL.
 - The ST20A finds a conditioning beaker after the second sample beaker. It then conditions and rinses according to the defined parameters.
 - After the fourth sample beaker the ST20A **must** condition. If it finds no conditioning beaker, it searches for one by turning backwards (see Note 2b.). It conditions for 30 s and rinses 5 mL.

- c. If you would like to condition at liberty during the run, but for a certain time and with a defined rinse volume,
- add a **Conditioning** function to the method and
 - set the interval to **60**, for example, and set the time and rinse volume.

The ST20A **must** not condition during the sample series if this is done. (Note: you can not designate more than 60 samples to a method).

2.3.11 Auxiliary instrument

You can use this function to control an auxiliary instrument (dispenser, relay, electromagnetic valve, etc.) that is attached to a 24 V auxiliary output of the titrator.

1. Select the name of the auxiliary instrument from the recommendation menu or enter one you have installed (see Section 1.5).
2. Enter the time [s]: It indicates how long the voltage should be applied at the auxiliary output. A formula may be entered instead of a number, for example, "H5/2" (H5 is a time increment which has been saved as an auxiliary value, see Section 1.6).
3. Select a condition:
 - No.
 - Yes: Enter the condition.

2.3.12 Titration

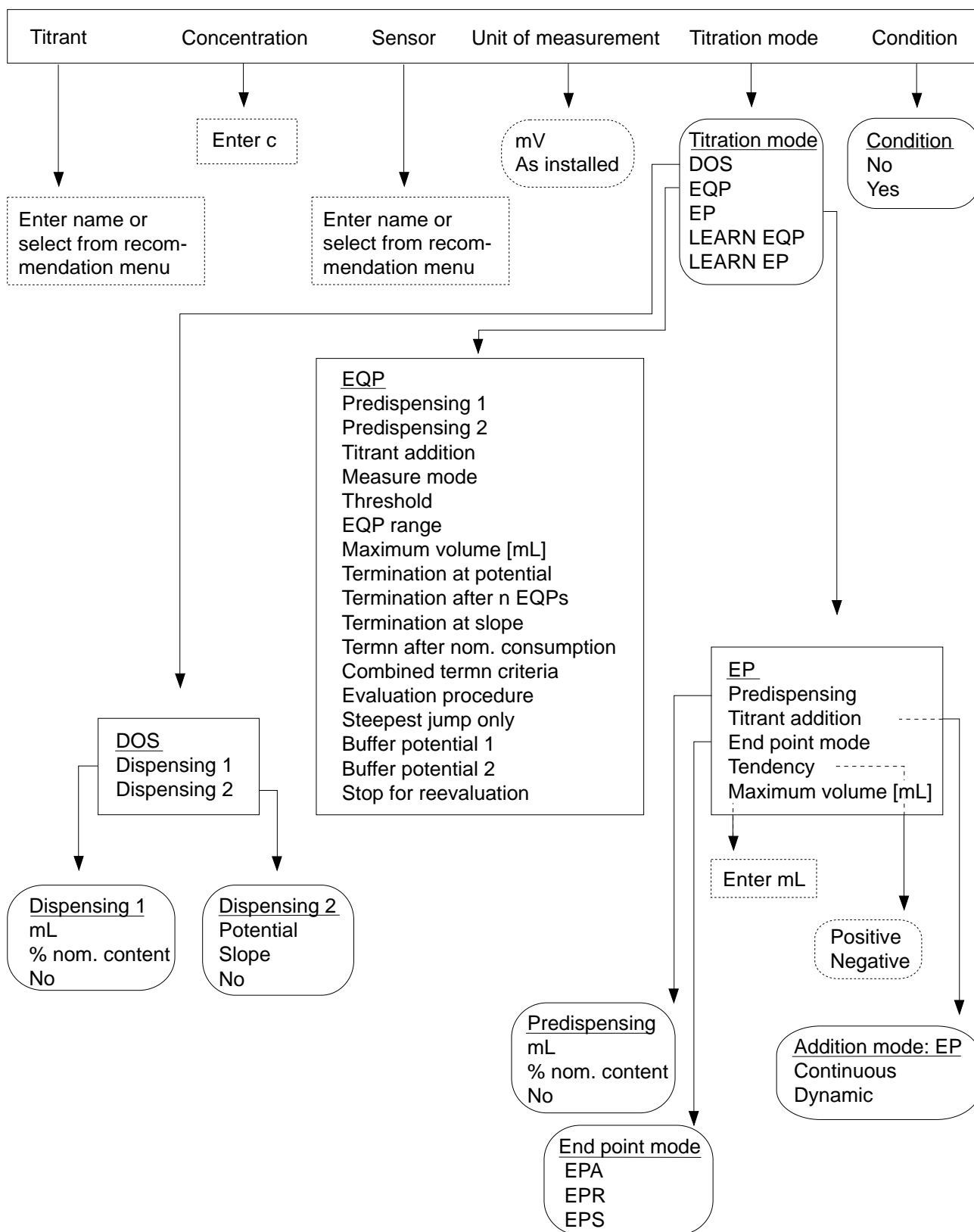
Under this function you determine the mode, control and evaluation of a titration. Depending on the titration mode, the titrator acquires several measured values and volumes as raw results (see *List of designations*, Section 8.1 and *Measure* function, Section 2.3.4).

If you have selected a temperature sensor in the **Sample** function, the temperature of the sample solution is measured automatically before the titrator executes this function. If no sensor is attached (parameter "Manual"), the titrator adopts the temperature entered at the start of the method. The slope of the pH electrode is then corrected for the temperature in the pH value calculation (see Section 4.7: *Calibration of the temperature sensors*).

Parameters of the mask:

Titrant
Concentration [mol/L]
Sensor
Unit of meas.(urement)
Titration mode
Condition

1. Select the titrant from the recommendation menu or enter one you have installed (see Section 1.1).
2. Enter its concentration [mol/L].
3. Select the sensor from the recommendation menu or enter one you have installed (see Section 1.2).
4. Select the unit of measurement: "mV" or "As installed".
"As installed" refers to the unit of measurement you have specified for the sensor (see Section 1.2.2).
5. Select the titration mode from the selection menu.
DOS (Dispensing)
EQP (Equivalence point titration)
EP (End point titration)
LEARN EQP (Learn titration: equivalence point determination)
LEARN EP (Learn titration: end point determination)
6. Select a condition:
 - No.
 - Yes: Enter the condition.

Titration: Menu tree

2.3.12.1 DOS (Dispensing)

In this titration mode the titrator acquires not only the raw results mL (VEQ) or mmol (Q) but also the potential values ET1 and ET2 or ET3 before and after dispensing. The titrator recognizes four different dispensing modes.

Selection menu Dose 1

You can dispense a specified volume. You either enter the number of mL directly or you let the titrator calculate this if you know the nominal content of the sample to be titrated.

1. Select mL and enter the volume.
A formula, such as "VDISP * 1.2", can be entered instead of a number (VDISP is the volume dispensed in a preceding **Dispense** function).
2. Select % nominal content (see Section 8.6.3); you must enter the following values here:
 - the metered amount in % of the nominal consumption
 - the nominal content
 - the conversion constant You can select this constant from the recommendation menu.
 - the maximum volume [mL]. It is intended as a safeguard: If, for instance, the wrong constant has been selected excess titrant is not dispensed needlessly.

If you press the **HELP** key (selector bar is positioned on *Conversion constant*) the titrator lists the constants with the corresponding units.

3. No: You do not need this dispensing mode.

Note: With these dispensing parameters the titrator waits for establishment of an equilibrium in the solution before it acquires the initial potential ET1. It then dispenses and after reestablishment of equilibrium acquires ET2.

Selection menu Dose 2

You can dispense either to a specified potential or a specified slope of the titration curve.

1. When you select potential you obtain the following parameters:
 - Potential: Enter the potential [mV, pH, ...].
 - Maximum volume [mL]: Enter the volume. It is intended as a safeguard: If, for instance, the wrong parameters have been selected excess titrant is not dispensed needlessly.

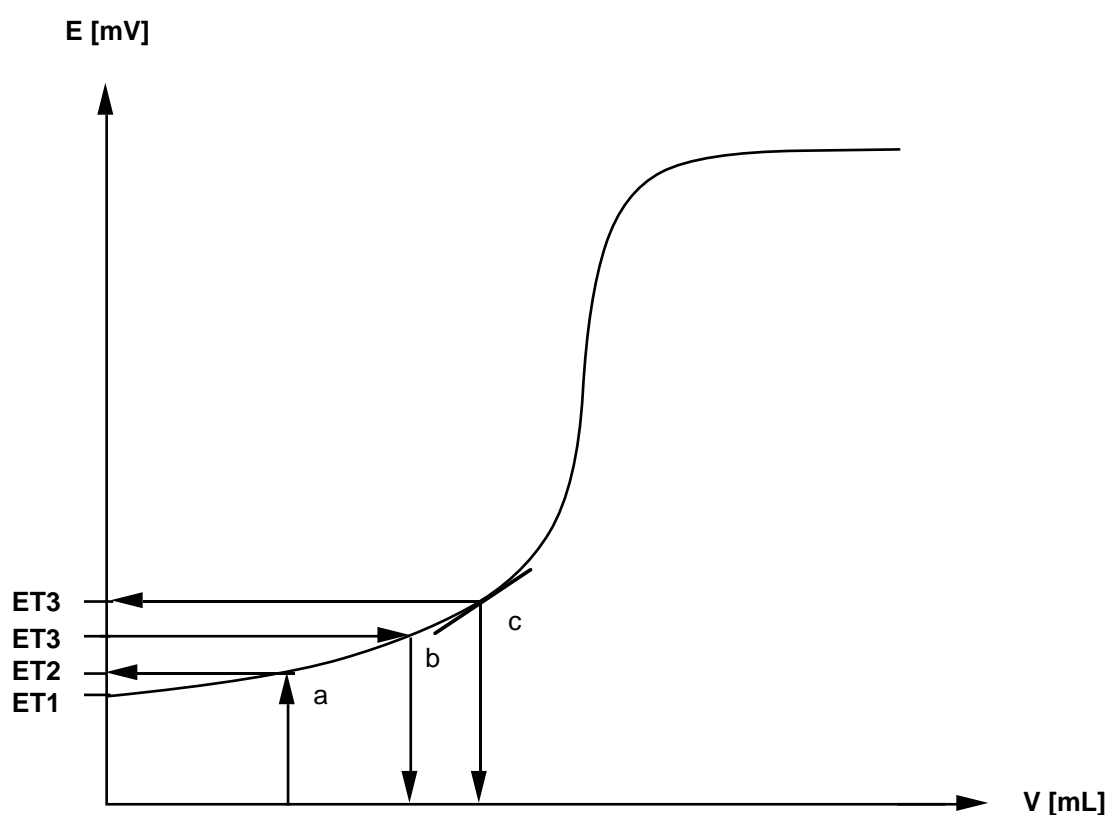
2. When you select the slope you obtain the following parameters:

- Slope: Enter the slope [mV, pH,.../mL].
- Maximum volume [mL]: Enter the volume (see point 1).

3. No: You do not need this dispensing mode.

Notes: a. With these dispensing parameters the titrator dispenses dynamically with equilibrium-controlled acquisition of the measured value (see representation under Section 2.3.12.2: *Titration mode EQP: Measure mode EQU*): It acquires the potential values ET1 and ET3.

b. You can link Dose 1 with Dose 2.



a: mL or nominal content dispensing

b: dispensing to a potential

c: dispensing to the slope of the curve

2.3.12.2 EQP (Equivalence point titration)

The equivalence point is the point at which exactly the same number of equivalents of titrant and analyte have reacted. In most cases it is virtually identical to the inflection point of the titration curve. This inflection point is recognized (EPOT = equivalence point potential) and the equivalence point calculated (VEQ or Q = mL or mmol consumption up to the equivalence point).

With this titration mode the titrator also determines the half neutralization value EHNV as a raw result.

Parameters of the mask:	Predispensing 1
	Predispensing 2
	Titrant addition
	Measure mode
(Equivalence point recognition):	Threshold
.....	EQP range
(Termination criteria):	Maximum volume [mL]
.....	Termination at potential
.....	Termination after n EQP's
.....	Termination at slope
.....	Termn (ination) after nom.(inal) consumption
.....	Combined termn (ination) criteria
(Evaluation criteria):	Evaluation procedure
.....	Steepest jump only
.....	Buffer potential 1
.....	Buffer potential 2
.....	Stop for reevaluation

A **predispensing** shortens the titration time. The titrator recognizes four predispensing modes.

Selection menu Predispensing 1

Under this selection menu you can predispense a certain volume. You either enter the number of mL directly or you let the titrator calculate this if you know the nominal content of the sample to be titrated.

1. Select mL and enter the volume.
A formula, such as "V DISP * 1.2", can be entered instead of a number (V DISP is the volume dispensed in a preceding **Dispense** function).
2. Select % nominal content (see Section 8.6.3); you must enter the following values:
 - the metered amount in % of nominal consumption
 - the nominal content
 - the conversion constant. You can select this constant from the recommendation menu.

If you press the **HELP** key (selector bar is positioned on *Conversion constant*) the titrator lists the constants with the corresponding units.

3. No: You do not need this kind of predispensing.

Note: The titrator dispenses the titrant in three steps (4/7, 2/7, 1/7 of the specified volume), which allows optimum calculation of the volume increment added when a dynamic titration follows. It acquires the potential values ET1 and ET2 (see titration mode *DOS*, Section 2.3.12.1).

Selection menu Predispensing 2

You can predispense to either a specified potential or a specified slope of the titration curve.

1. Enter the potential [mV, pH, ...].
2. Enter the slope [mV, pH, .../mL].
3. No: You do not need this kind of predispensing.

Notes: a. The titrant addition in this predispensing mode follows the selected parameters of the main titration, however, the increments are greater. The titrator also registers the measured values quicker and acquires the potential values ET1 and ET3 (see titration mode *DOS*, Section 2.3.12.1).

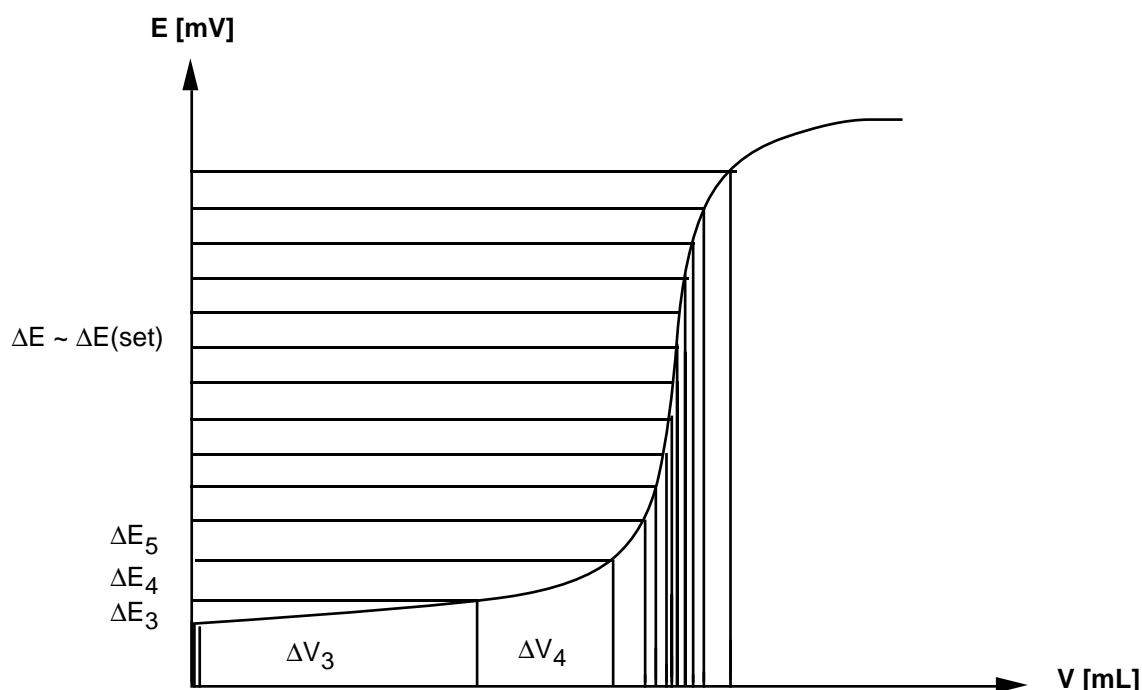
- b. You can link Predispensing 1 with Predispensing 2.

Selection menu Titrant addition

You can select the dynamic (DYN) or incremental (INC) addition mode. You can not decide the optimum addition mode for your titration until you know what type of titration curve you have.

DYN

The volume increment added by the titrator changes within the defined limits $\Delta V(\text{min})$ and $\Delta V(\text{max})$. This should lead to a constant potential difference ΔE per increment.



1. Enter $\Delta E(\text{set})$ [mV]: e.g. 10.
2. Select the limits ΔV and enter $\Delta V(\text{min})$ (smallest increment) and $\Delta V(\text{max})$ (largest increment) as absolute or relative values:
 - a. Absolute
 - Enter $\Delta V(\text{min})$ [mL]: e.g. 0.05 (see following page).
 - Enter $\Delta V(\text{max})$ [mL]: e.g. 0.3.

Notes: a. The smallest increment, that the titrator can add, is 1/5'000 of the burette volume:

1 mL burette → 0.0002 mL

5 mL burette → 0.001 mL

10 mL burette → 0.002 mL

20 mL burette → 0.004 mL

- b. If no predispensing takes place, the titrator adds the first two volume increments with $\Delta V(\text{min})$.

2 b. Relative

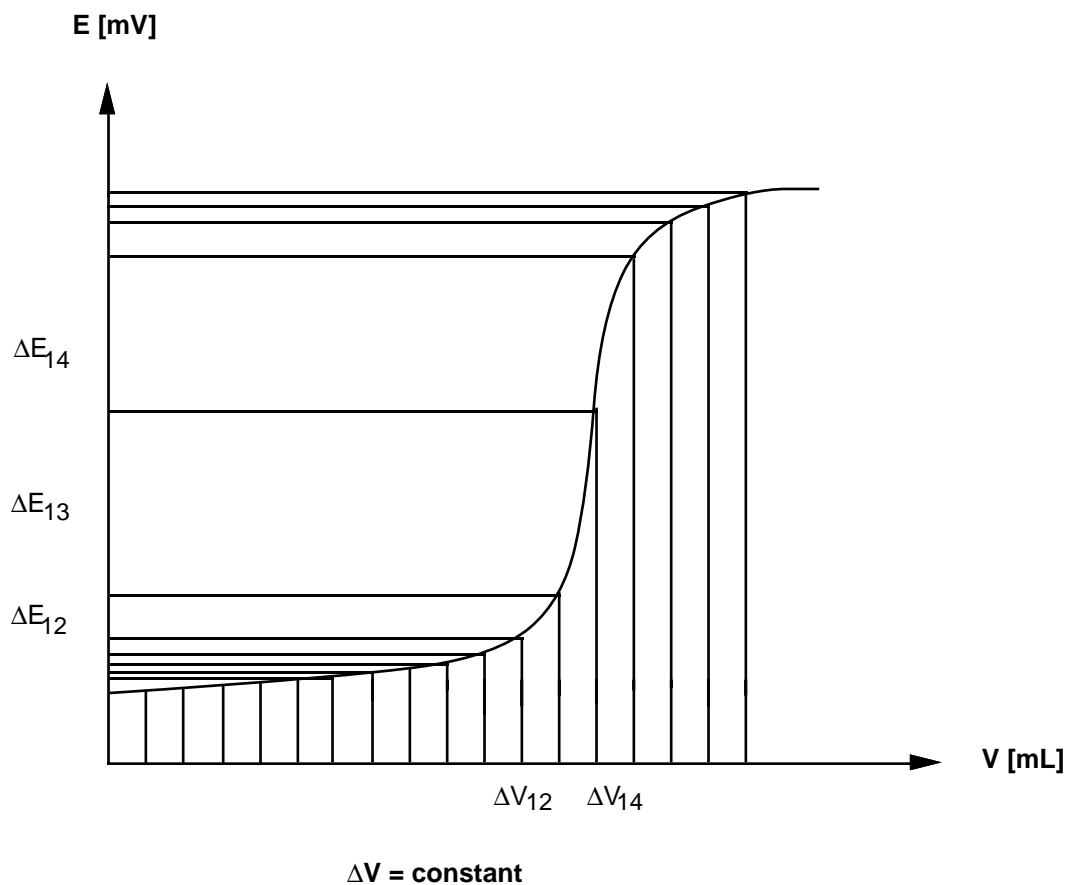
- Enter $\Delta V(\text{min})$ [%dosVol]: refers to the volume already dispensed, in other words the minimum set increment increases during the course of the titration.
- Enter $\Delta V(\text{max})$ [%buVol]: refers to the burette volume and remains constant.

Note: If no predispensing takes place, the titrator calculates and dispenses first two volume increments relative to 10% of the burette volume.

INC

The volume increment added by the titrator remains constant.

1. Enter ΔV [mL], e.g. 0.1.



Selection menu Measure mode

You can select the equilibrium controlled (**EQU**) or time controlled (**TFIX**) measured value acquisition. This determines the waiting time between addition of the increments. Using **EQU** the waiting time is variable, using **TFIX** it is constant. You can not decide the optimum measure mode for your method until you know the reaction time of the components and the response time of the sensor used.

EQU

Before the titrator accepts a measured value, equilibrium must be established in the solution.

The following parameters are responsible for the equilibrium

ΔE potential change of the solution

Δt within a time

Time limits between the increments are the parameters

t(min) minimum time

t(max) maximum time

As soon as the potential change of the solution is smaller than the specified equilibrium ($\Delta E/\Delta t$), the titrator will acquire the measured value and add the next increment.

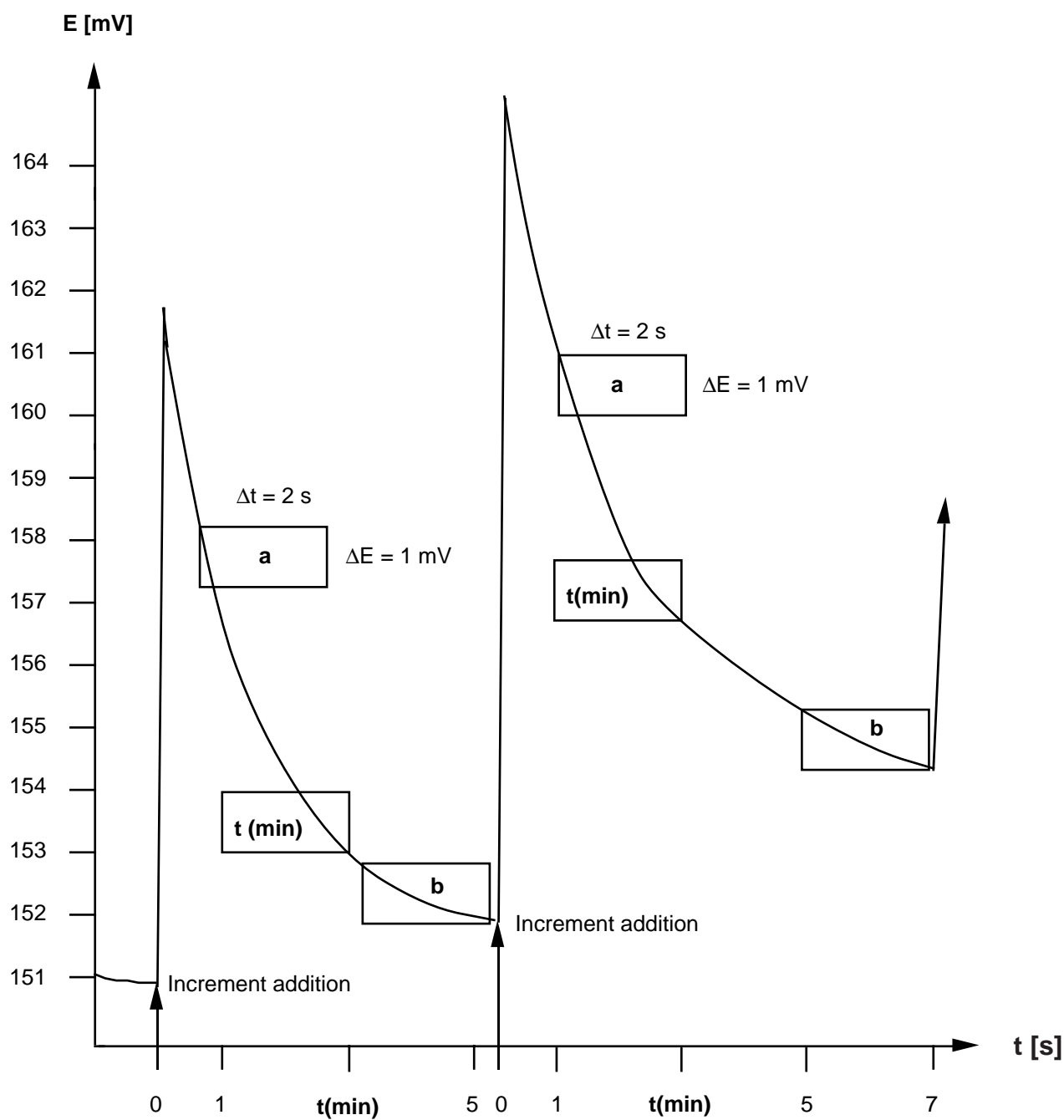
This **can** be established at the earliest at t(min) and **should** be established at the latest at t(max). At t(max) the measured value is acquired at all events even if the equilibrium condition has not yet been met.

Examples for equilibrium conditions

	fast titrations (acid-base titrations in aqueous media)	slow titrations (precipitation titrations in non-aqueous media)
1. Enter ΔE [mV]:	1	0.5
2. Enter Δt [s]:	1	2
3. Enter t(min) [s]:	3	4
4. Enter t(max) [s]:	15	30

Notes: a. When measuring in **EQU**, increment addition is rapid in the flat segment of the titration curve and slow in the steep segment.

b. The titrator acquires the time and the titrant volume together with the potential. These measured values can be printed **after** the sample has been titrated (see Section 2.3.19: *Record*).



a: The defined equilibrium condition has not been satisfied.

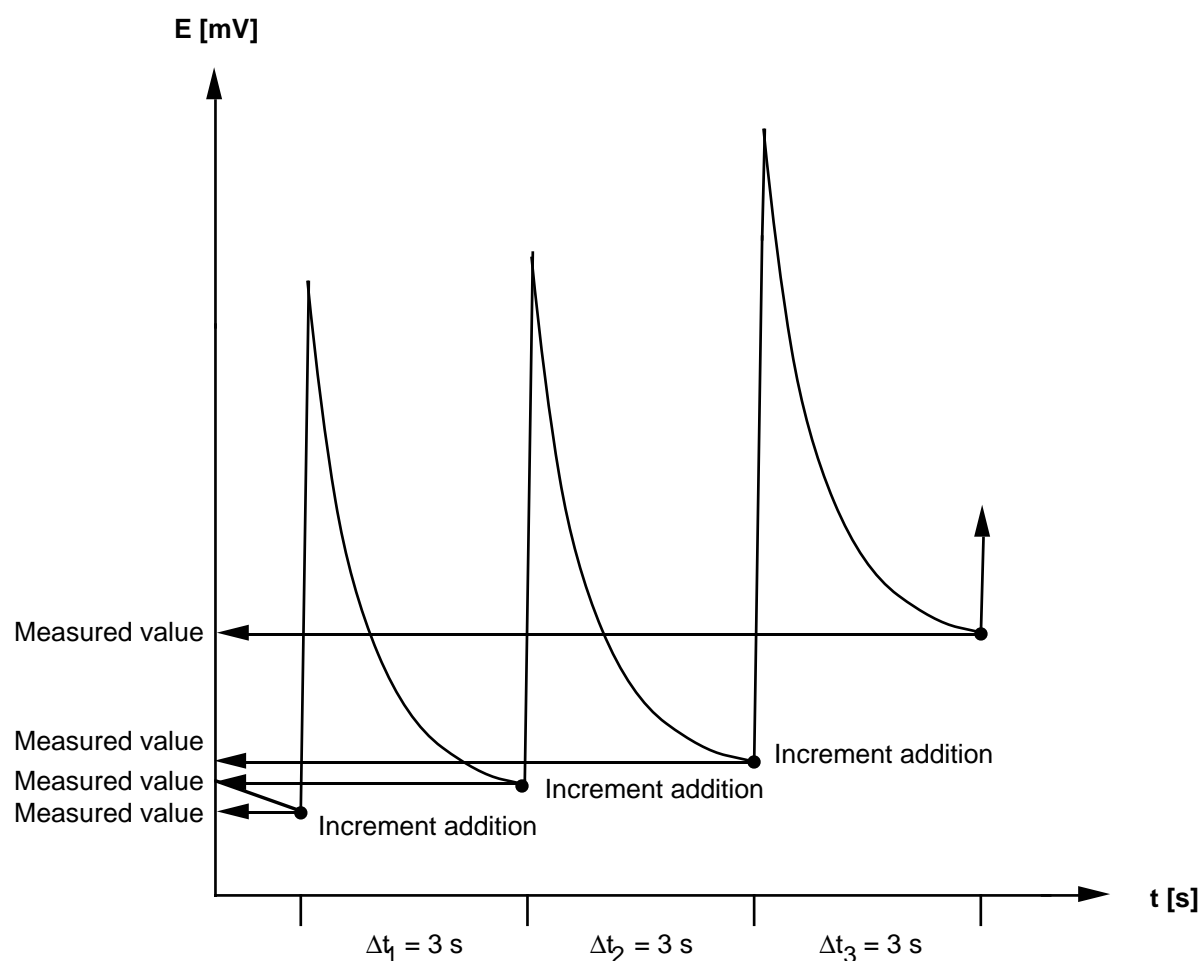
t(min): The equilibrium condition has not been satisfied after 3 s.

b: The equilibrium condition has been satisfied after 5.4 or 6.9 s for the first time.

TFIX

After each increment addition the titrator waits for the time you have defined before it accepts the measured value.

1. Enter Δt [s].



Equivalence point recognition

The equivalence point recognition with a titration curve depends on the reaction type of the components and hence on the evaluation procedure (see below this section).

1. In the evaluation procedures **Maximum** and **Minimum** an equivalence point is recognized when the highest (lowest) potential value of the titration curve is greater (smaller) than two preceding and two subsequent values.
2. In the evaluation procedures **Standard** and **Asymmetric** an equivalence point is recognized when the maximum of the absolute values of the 1st derivative of the titration curve is greater than two preceding and two subsequent values.
3. In the evaluation procedure **Segmented** an equivalence point is recognized when the maximum of the absolute values of the 2nd derivative of the titration curve is greater than two preceding and two subsequent values.

Note: For the earliest possible recognition of an equivalence point, the titrator requires a certain number of measurement points.

- The number depends on the evaluation procedure.
- Measurement points from a predisensing are not considered!

Evaluation procedure	Earliest possible EQP at	Measurement points necessary
Minimum/Maximum	4th measurement point	6
Standard	4th measurement point	6
Asymmetric	4th measurement point	10
Segmented	5th measurement point	8

An example for the evaluation procedure **Minimum**: If the third measurement point lies in the vicinity of the lowest potential value, it will not be recognized as an equivalence point.

You must or can assist the recognition using two parameters.

Threshold

To prevent small disturbances in the curve being identified as an equivalence point, you **must** specify a threshold value: ([+/-mV, pH...], [mV, pH.../mL] or [mV, pH.../mL²]). This must be exceeded.

Note: a. The maximum value of the threshold should not exceed half the expected maximum value of the first or second derivative at the equivalence point.

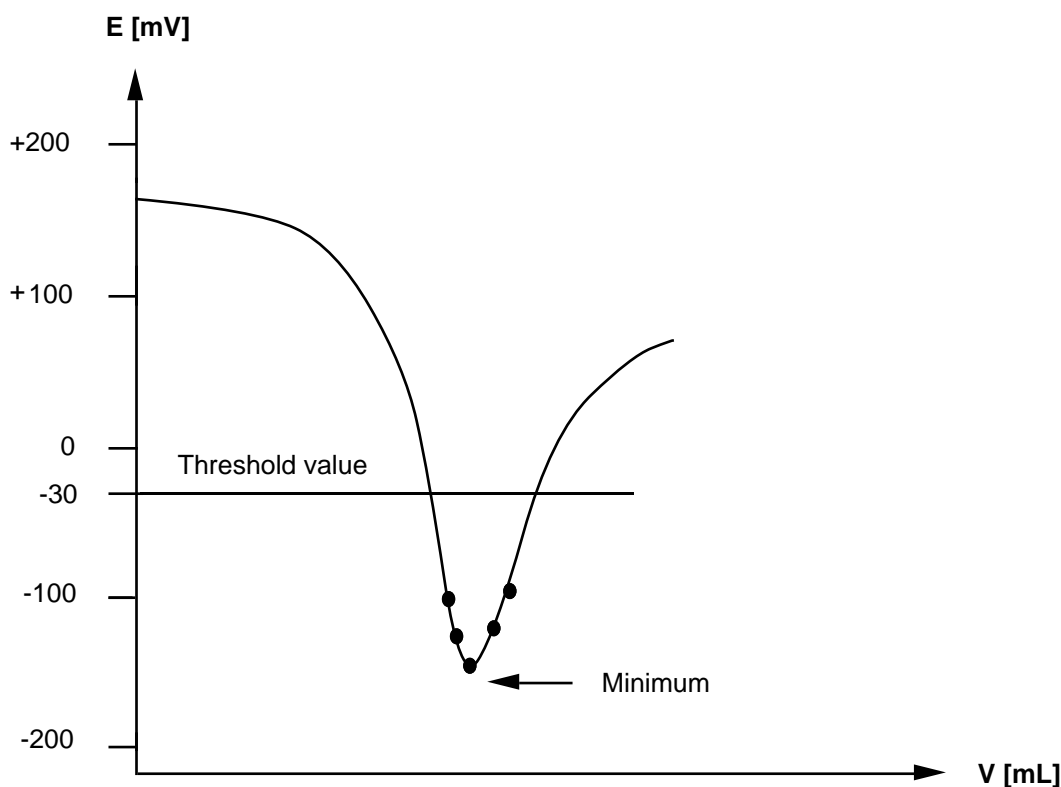
Note: b. The maximum or minimum, the maximum of the absolute values of the 1st derivative and the maximum of the absolute values of the 2nd derivative of a titration curve all depend on so many factors (solvent, concentration, sensor, type of reaction, etc.) that you can not define the "right" threshold value until you have performed the first titration.

You can enter one of the following values for this:

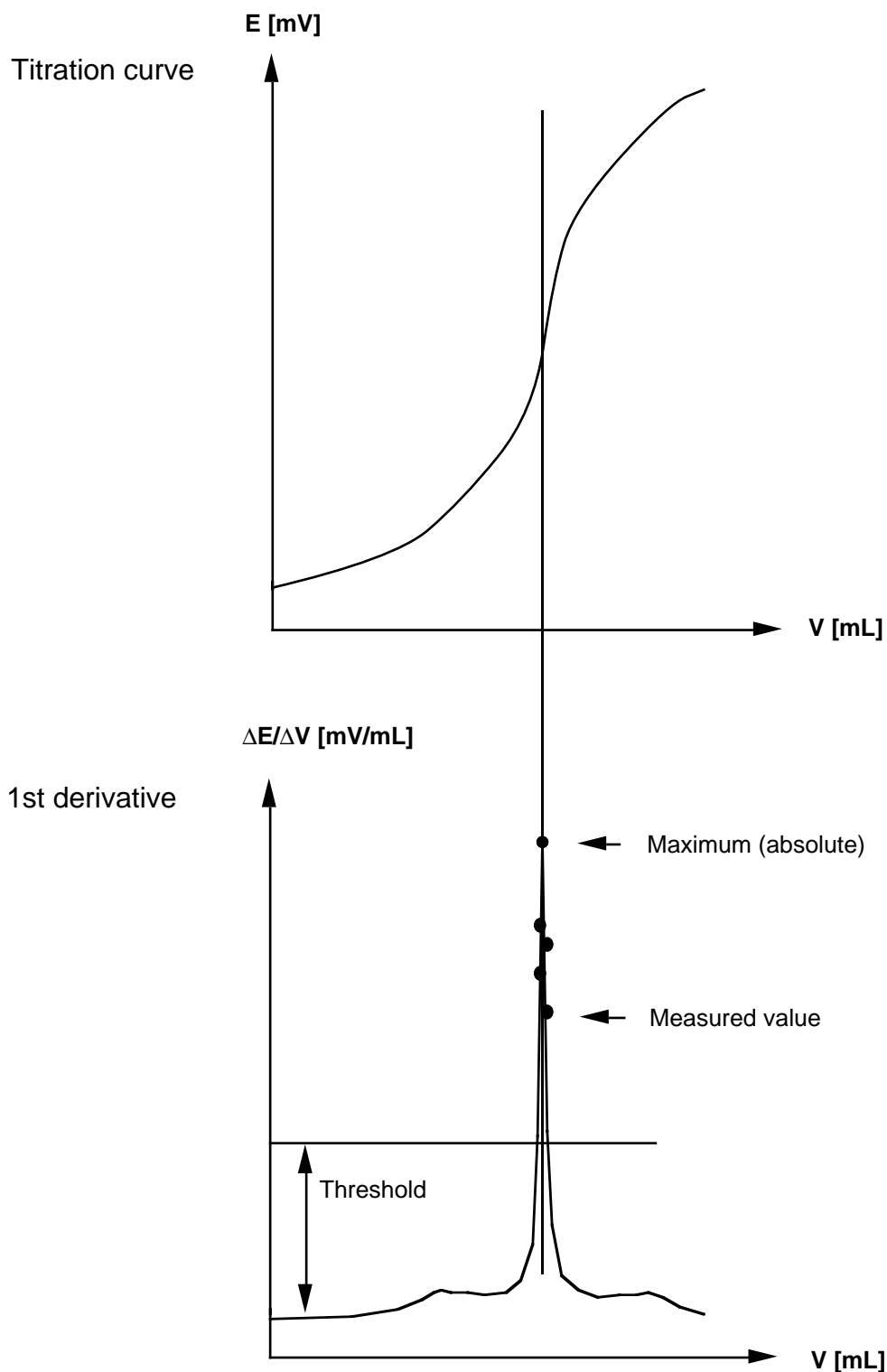
Evaluation procedure	mV	pH/pM/pX	%T
Minimum/Maximum	0	0	0
Standard/Asymmetric	10	0.2	1
Segmented	10	0.2	1

With the aid of the appropriate titration curve or table of measured values (there is no table for the 2nd derivative) you can read off the potential value, the values for $\Delta E/\Delta V$ or the values for $\Delta^2 E/\Delta V^2$ and then enter the threshold value.

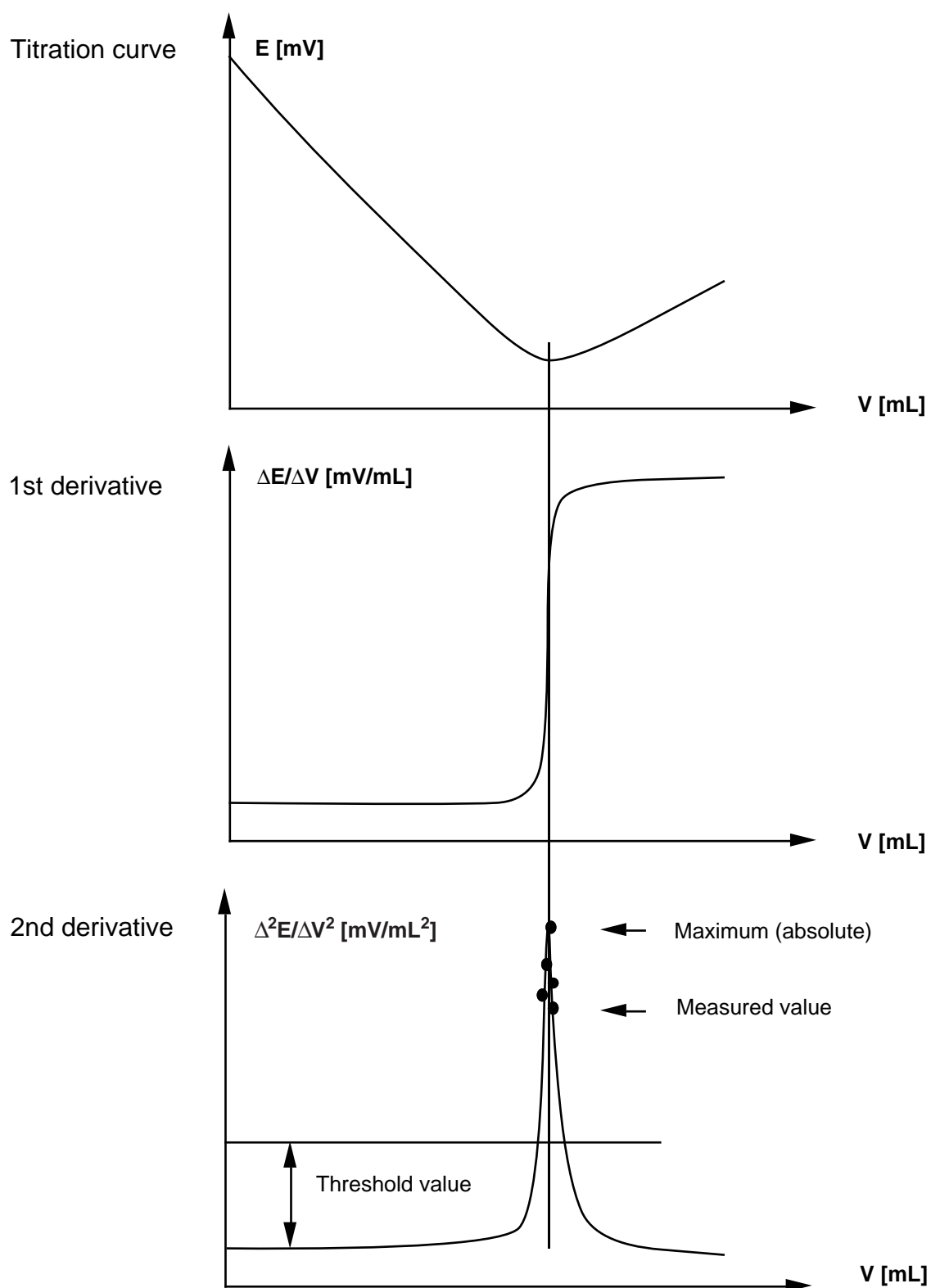
1. Example of the threshold value of a titration curve for the evaluation procedure **Minimum**:
The minimum of this titration curve is at -150 mV. You can enter, for instance, **-30** as threshold value. The sign for the mV value must also be entered.



2. Example of the threshold value of a titration curve for the evaluation procedures **Standard** and **Asymmetric**:



3. Example of the threshold value of a titration curve for the evaluation procedure **Segmented**:



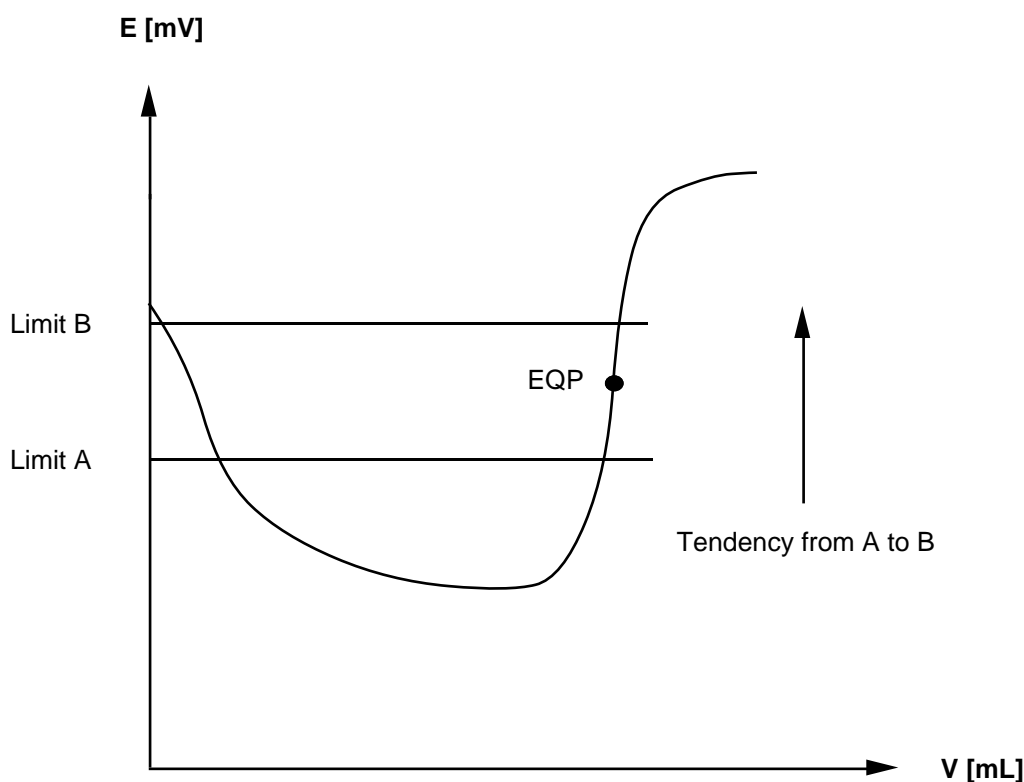
EQP range

To identify the equivalence point you can also define a potential range. For this you enter two potential values (the order in which you enter them defines the tendency) to specify the part of the titration curve – falling or rising – where the equivalence point should be located. Equivalence points that lie outside this EQP range or which exhibit the opposite tendency are not registered.

– No.

– Yes: Enter limit A [mV, pH,...].
Enter limit B [mV, pH,...].

A formula, such as " $H_2 + 50$ ", or a potential stored as an auxiliary value can be entered instead of a number (see *Auxiliary values*, Section 1.6 and *Auxiliary value* function, Section 2.3.15).



Note: The EQP range refers to the titration curve, i.e. it is independent of the evaluation procedure.

Termination criteria

You can determine when a titration should be terminated by selecting six different parameters. The titrator can abort the titration, either

- after the **first** of the selected criteria is satisfied or
- after **all** selected criteria are satisfied.
- An exception is the maximum volume: the titration is always terminated immediately once this has been reached!

Maximum volume

You must enter the maximum volume [mL]. It is intended as a safeguard: If the titration is faulty excess titrant is not dispensed needlessly as the titration is always aborted.

Select **Termination at potential**:

The titrator terminates the titration at the specified potential.

– No.

– Yes: Enter the potential [mV, pH, ...].

A potential stored as an auxiliary value **Hj** or a formula can be entered instead of a number (see *Auxiliary values*, Section 1.6 and *Auxiliary value* function, Section 2.3.15).

Select **Termination after n EQPs**:

The titrator terminates the titration after a certain number of equivalence points **n** have been found.

– No.

– Yes: Enter n.

Select **Termination at slope**:

The titrator terminates the titration when the slope of the titration curve falls below a specified value. The slope of the curve must exceed this absolute value once then fall below it twice to lead to termination.

– No.

– Yes: Enter the slope [mV, pH, .../mL].

Select **Termination after nominal consumption**:

The titrator terminates the titration when the added volume is, e.g. 10% above the nominal consumption up to the equivalence point (see Section 8.6.3).

– No.

– Yes: Enter the termination volume in % of the nominal consumption.

Enter the nominal content.

Enter the conversion constant or select it from the recommendation menu.

If you press the **HELP** key (selector bar is positioned on *Conversion constant*) the titrator lists the constants with the corresponding units.

Select Combined termination criteria:

The titration is terminated only after all the specified criteria have been fulfilled (exception: maximum volume, see above).

Evaluation criteria

You determine which type of calculation should be used to calculate the equivalence point found and select the jump of a titration curve or potential values that you wish to have evaluated. You can combine these parameters.

Evaluation procedures

You can choose the procedure which is suitable for the titration curve from several calculation modes (see Section 8.4).

Standard	Evaluation procedure for all S-shaped titration curves
Asymmetric	Evaluation procedure for S-shaped, highly asymmetric titration curves
Segmented	Evaluation procedure for titration curves with individual sections (segmented curve)
Minimum	Determination of the minimum of a titration curve
Maximum	Determination of the maximum of a titration curve

Select Steepest jump only:

- No.
- Yes: Only the steepest jump of the titration curve is evaluated by the titrator.

Select Buffer potential 1:

When a buffer potential is selected the titrator not only evaluates any equivalence points present but also the titrant consumption in mmol (QP1, QP2) or mL (VP1, VP2) up to the attainment of these potential values (see *List of designations*, Section 8.1).

- No.
- Yes: Enter buffer potential 1 [mV, pH, ...].
A potential stored as an auxiliary value **Hj** or a formula can be entered instead of a number (see *Auxiliary values*, Section 1.6 and *Auxiliary value* function, Section 2.3.15).

Select Buffer potential 2:

- No.
- Yes: Enter buffer potential 2 [mV, pH, ...] (see buffer potential 1).

Caution: If you have selected P1 and/or P2, the titrator checks all termination criteria – except maximum volume – only when the appropriate buffer potentials P1 and/or P2 have been reached.

With the selection of both buffer potentials, you also define the tendency (from P1 to P2) and the order: If the titrator first finds P2, it will no longer search for P1.

Select Stop for reevaluation:

The **Titration** function is interrupted during the run as soon as termination criteria and the specified condition are fulfilled. This enables you to modify parameters for the equivalence point recognition (threshold value, EQP range) and for the evaluation (buffer potentials P1 and P2). All data will be reevaluated with the modified parameters (see Section 3.5).

- No.
- Yes: Enter a condition, for example "neq = 0" (no equivalence point found), see *Functions with a condition*, Section 8.3.

2.3.12.3 EP (End point titration)

With this titration mode you titrate to a specified value of the selected unit of measurement.

Caution: Before an end point titration you should calibrate the appropriate sensor!

Parameters of the mask:	Predispensing
	Titrant addition
	End point mode
	Tendency
	Maximum volume [mL]

Selection menu Predispensing

Predispensing shortens the time of titration: Under this selection menu you can predispense a defined volume. You either enter the number of mL directly or you let the titrator calculate this if you know the nominal content of the sample to be titrated.

1. Select mL and enter the volume.
A formula, such as "VDISP * 1.2", can be entered instead of a number (VDISP is the volume dispensed in a preceding **Dispense** function).
2. Select % nominal content (see Section 8.6.3); here you must enter the following values:
 - the metered amount in % of the nominal consumption
 - the nominal content
 - the conversion constant. You can select this constant from the recommendation menu.

If you press the **HELP** key (selector bar is positioned on *Conversion constant*) the titrator lists the constants with the corresponding units.

3. No: You need no predispensing.

- Notes:
- a. With subsequent continuous titrant addition, the titrator dispenses the volume in one step. It acquires the potential values ET1 and ET2 (see Section 2.3.12.1: *Titration mode DOS*).
 - b. With subsequent dynamic titrant addition, the titrator dispenses the volume in three steps (4/7, 2/7, 1/7 of the specified volume), which allows optimal calculation of the increment to be added. It acquires the potential values ET1 and ET2.

Selection menu Titrant addition

You select the continuous or dynamic titrant addition.

Continuous

The titrator dispenses the titrant slowly at first then at maximum rate up to a specified control band [mV, pH,...]. Within the control range the rate diminishes exponentially. In the vicinity of the end point it adds the increment that you define (the smallest increment that the titrator can dispense is 1/5'000 of the burette volume).

It measures the initial potential ET1 after 1-3 s and acquires a measured value every 5 seconds for the table of measured values.

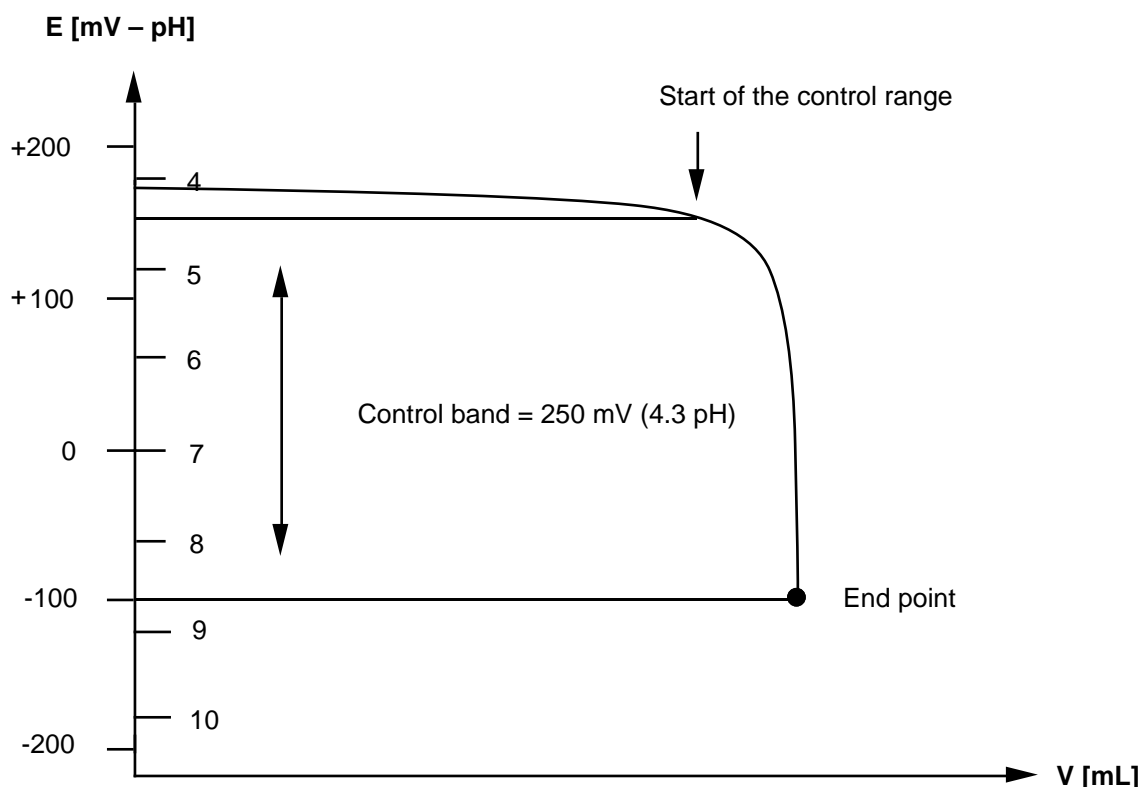
1. Enter control band [mV, pH, ...]: e.g. 250.

2. Enter delay [s]: e.g. 15.

This is the time from attainment of the end point up to the definitive termination of the titration. If the potential falls below that of the end point during this time, the titrator adds additional increments.

3. Enter $\Delta V(\text{min})$ [mL] : e.g. 0.01.

This is the smallest increment, to be added near the titration end point.



Dynamic

The conditions that apply to the dynamic titrant addition are the same as those for **DYN** of the equivalence point titration **EQP** (see Section 2.3.12.2).

1. Enter $\Delta E(\text{set})$ [mV]: e.g. 10.

2. Enter $\Delta V(\text{min})$ [mL] (smallest volume increment): e.g. 0.05.

3. Enter $\Delta V(\text{max})$ [mL] (largest volume increment): e.g. 0.3.

If no predispensing is performed, the titrator dispenses the first two volume increments with $\Delta V(\text{min})$.

The measured value acquisition is equilibrium controlled (see Section 2.3.12.2: *Titration mode EQP: Measure mode EQU*). You must therefore enter the following parameters:

4. ΔE [mV] : e.g. 1.

5. Δt [s] : e.g. 2.

6. $t(\text{min})$ [s] : e.g. 3.

7. $t(\text{max})$ [s] : e.g. 30.

This equilibrium condition applies only in the end point range defined by the following formula:

$$\text{EP range} = \text{EP} \pm \{1.5 * \Delta E(\text{set})\}.$$

Example: If the end point is -30 mV, the condition thus applies for above values of -15 to -45 mV.

Outside this range the following applies: $\Delta E(\text{outside}) = 4 * \Delta E$.

If the endpoint is reached, the following applies: $\Delta E = \Delta E/2$.

8. Enter delay [s] : e.g. 15.

This is the time from attainment of the end point up to the definitive termination of the titration. If the potential falls below that of the end point during this time, the titrator adds additional increments.

Selection menu End point mode

You have a choice between three end point titrations.

1. Select **EPA** (absolute end point):

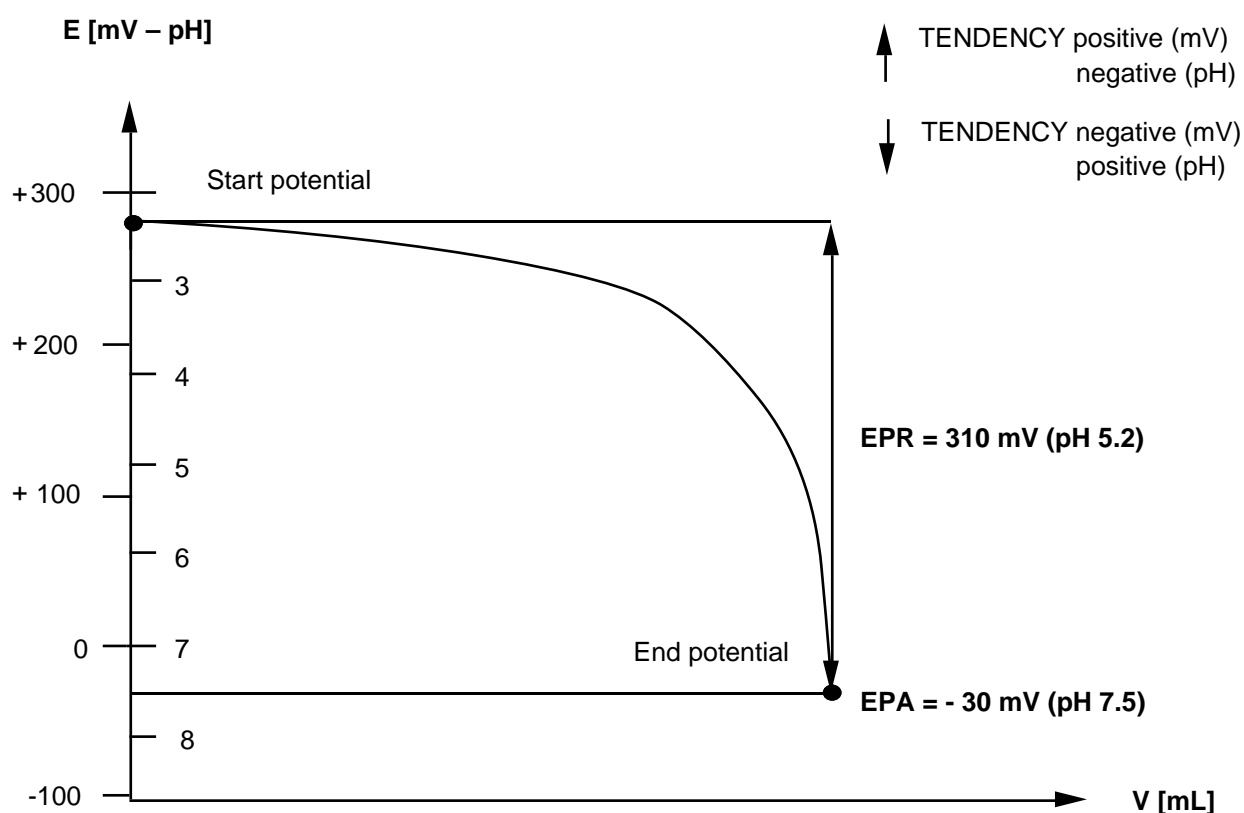
The absolute end point is the end point value relative to zero on the electrode signal scale.

– Enter EPA (mV, pH, ...).

2. Select **EPR** (relative end point):

The relative end point is the difference between the electrode signal at the start and end of the titration.

– Enter EPR (mV, pH, ...).



3. Select **EPS** (other end point):

– Enter the end point, e.g. H2:

This end point can be stored as auxiliary value **Hj** and you can call it up here (see *Auxiliary value* function, Section 2.3.15 and *Auxiliary values*, Section 1.6).

Example: Assuming you have stored the value of "EPOT of H_3PO_4 at 1st equivalence point" under **H2** and you would now like to titrate to this potential, enter **H2**.

You can also enter a formula, such as " $E + 200$ " (E is the measured potential of a preceding **Measure** function).

Tendency

A titration exhibits a positive or negative potential or pH change (see illustration). This has to be specified by you.

- Select "Positive" or "Negative".

Caution: A positive potential change means a negative pH change!

Maximum volume

You must enter the volume [mL]. It is intended as a safeguard: If the titration is faulty excess titrant will not be dispensed needlessly.

2.3.12.4 LEARN EQP (Learn titration: equivalence point determination)

If you are not sure what parameters you should enter for the titration mode EQP, select **LEARN EQP**.

Titration function: selection of the parameters (example)

Titrant	AgNO ₃
Concentration [mol/L]	0.01
Sensor	DM140-SC
Unit of meas.(urement)	As installed
Titration mode	LEARN EQP
Condition	No

When you start the method, the titrator executes one function after the other. As soon as it has completed the **Titration** function, it calculates the parameters for the titration mode EQP, **stores** them and sends the data to the printer. While the parameters of the **Titration** function are being printed, the titrator executes the remaining functions.

Printout of a learned EQP titration.

LEARN TITRATION

Method	1e2		
	Version	30-Sep-1992	10:14

Titration [1]

Titrant	AgNO ₃
Concentration [mol/L]	0.05
Sensor	DM141-SC
Unit of meas.	As installed
Titration mode	EQP
Predispensing 1	mL
Volume [mL]	0.5
Predispensing 2	To potential
Potential [mV, pH, ...]	-16.5
Titrant addition	DYN
ΔE(set) [mV]	8.0
Limits ΔV	Relative
ΔV(min) [%dosVol]	0.5
ΔV(max) [%buVol]	4.0
Measure mode	EQU
ΔE [mV]	0.5
Δt [s]	2.0
t(min) [s]	20.0
t(max) [s]	100.0
Threshold	10.0
Maximum volume [mL]	10
Termination after n EQPs	Yes
n =	1
Evaluation procedure	Standard

The titrator always titrates the entire volume of the burette used. Thus

- you must select the weight/volume of your sample in accordance with the burette volume.

The titrator calculates the parameters from the response behavior of the sensor, the shape of the titration curve and with due consideration of the burette volume used. You thus always obtain

- the **relative limits** $\{\Delta V(\min), \Delta V(\max)\}$ of the dynamic titrant addition **DYN** and
- the parameters of the measure mode **EQU**.

If the titrator finds more than one equivalence point, it calculates the parameters for the one with the **steepest** jump.

If the titrator finds no equivalence point, it interrupts the method. You are shown an error message (see Section 3.4).

- Notes:
- a. Since the titrator immediately stores the parameters of the titration mode EQP, you can titrate a series of samples with the same content (**n > 1** in the **Sample** function). The statistics calculation then discards the result of the **first** sample!
 - b. If your method has several **Titration** functions, you can select **LEARN EQP** or **LEARN EP** for each titration mode.
 - c. The earliest possible equivalence point will be recognized if it lies in the vicinity of the **sixth** measurement point (measurement points from a pretitration are not considered, see *Equivalence point recognition*, page 2-45). If the equivalence point lies in the vicinity of the fifth point, for instance, it will not be recognized.

2.3.12.5 LEARN EP (Learn titration: end point determination)

If you are not sure what parameters you should enter for the titration mode EP, select **LEARN EP**.

Titration function: selection of the parameter (example)

Titrant	NaOH
Concentration [mol/L]	0.1
Sensor	DG111-SC
Unit of meas.(urement)	As installed
Titration mode	LEARN EP
Condition	No

When you start the method, the titrator executes one function after the other. As soon as it has completed the **Titration** function, it calculates the parameters for the titration mode EP, **stores** them and sends the data to the printer. While the parameters of the **Titration** function are being printed, the titrator executes the remaining functions.

Printout of a learned EP titration.

LEARN TITRATION

Method	Ber	LEARN TITRATION	
	Version	30-Nov-1992	12:53

Titration [1]

```

TitrantNaOH
Concentration [mol/L] ..... 0.1
Sensor ..... DG111-SC
Unit of meas. .... As installed
Titration mode ..... EP
  Predispensing ..... mL
    Volume [mL] ..... 1.0
  Titrant addition ..... Dynamic
    ΔE(set) [mV] ..... 8.0
    ΔV(min) [mL] ..... 0.05
    ΔV(max) [mL] ..... 0.15
    ΔE [mV] ..... 0.5
    Δt [s] ..... 0.5
    t(min) [s] ..... 2.0
    t(max) [s] ..... 20.0
    Delay [s] ..... 0
  End point mode ..... EPA
    Potential [mV, pH, ...] ..... 6.568
  Tendency ..... Positive
  Maximum volume ..... 20

```


The titrator always titrates the entire volume of the burette used. Thus

- you must select the weight/volume of your sample in accordance with the burette volume.

The titrator calculates the parameters from the response behavior of the sensor, the shape of the titration curve and with due consideration of the burette volume used. You thus always obtain

- the parameters of the titrant addition **Dynamic**
and
- the parameter of the endpoint mode **EPA**.

If the titrator finds more than one endpoint, it calculates the parameters for the one with the greatest slope.

The titrator always specifies its tendency at a detected endpoint.

If the titrator finds no endpoint, it interrupts the method. You are shown an error message (see Section 3.4).

Notes: a. Since the titrator immediately stores the parameters of the titration mode EP, you can titrate a series of samples with the same content (**n > 1** in the **Sample** function). The statistics calculation then discards the result of the **first** sample!

- b. If your method has several **Titration** functions, you can select **LEARN EQP** or **LEARN EP** for each titration mode.

2.3.13 pH/mV-stat

You use this function to perform a pH-stating. You determine the parameters which assure the constancy of a potential value, which lead to the termination of the pH-stating and which are used in the evaluation. The titrator acquires the following raw results (see *Measure* function, Section 2.3.4):

- The titrant consumption in mL (VTOT) or mmol (QTOT) up to termination of the pH-stating.
- The titrant consumption in mL (VT1, VT2) or mmol (QT1, QT2) up to the attainment of the specified time limits t1 and t2.
- The mean consumption in mL/min (VSTAT) or mmol/min (QSTAT) within the time limits t1 and t2 (see Section 8.1).
- The correlation coefficient CSTAT; this results from the calculation of the mean consumption through linear regression (see Section 8.1: *List of designations*).

If you have selected a temperature sensor in the **Sample** function, the temperature of the sample solution is measured automatically before the titrator executes this function. If no sensor is attached (parameter "Manual"), the titrator adopts the temperature entered at the start of the method. The slope of the pH electrode is then corrected for the temperature in the pH value calculation (see Section 4.7: *Calibration of the temperature sensors*).

Parameters of the mask:	Titrant Concentration [mol/L] Continuous addition Sensor Unit of meas.(urement) Pretitration End point (pH-stat) Control range [mV] Tendency
(Termination criteria):	Maximum volume [mL]
.....	t(min) [s]
.....	t(max) [s]
.....	Minimum consumption [mL]
.....	Time span [s]
(Evaluation criteria):	Time limit t1 [s]
.....	Time limit t2 [s]
(Data storage):	Time interval [s]
	Condition

1. Select the titrant from the recommendation menu or enter one you have installed (see Section 1.1).
2. Enter its concentration [mol/L].
3. Select continuous addition:

This parameter allows a pH-stating, in which more titrant than will fit in one burette volume is consumed, to be performed without interruption. For this purpose you use the same titrant. You must, however, rename it and install it on another drive (see Section 1.1.3). As soon as the contents of the first burette have been dispensed, the second begins dispensing. Thus the time required to refill the first burette can be bridged over.

 - No.
 - Yes: Enter the titrant "2" you have installed.
Enter its concentration [mol/L].
4. Select the sensor from the recommendation menu or enter one you have installed (see Section 1.2).
5. Select the unit of measurement: "mV" or "As installed".

"As installed" refers to the unit of measurement you have defined for the sensor (see Section 1.2.2).
6. Pretitration

You select the pretitration if your solution does not yet have the value of the potential you need for the pH-stating.

 - Yes: Enter control band [mV, pH, ...] (see Section 2.3.12.3: *Titration mode EP*).
 - No.
7. End point (pH-stat)

From the selection menu select the absolute or other end point and define the potential value that should be kept constant during the pH-stating.

 - EPA : Enter mV, pH, ... value (see Section 2.3.12.3: *Titration mode EP*).
 - EPS : Enter the end point (see Section 2.3.12.3: *Titration mode EP*).
8. Control range

As a control range you define a potential value that controls the end point range: The smaller this value, the faster the titrator reacts to a deviation from the potential value of the defined end point potential.

Enter control range [mV]: e.g. 2.
9. Select the tendency: "Positive" or "Negative" (see Section 2.3.12.3: *Titration mode EP*).

The choice of tendency refers to the added titrant. The actual reaction investigated by pH-stating always leads to a change in the opposite direction!

10. Enter the termination criteria:

- a. Maximum volume [mL]: e.g. 40. The entry is intended as a safeguard: If the titration is faulty excess titrant will not be added needlessly.
- b. Minimum time t(min) [s]: e.g. 600.
- c. Maximum time t(max) [s]: e.g. 1800.
- d. Minimum consumption [mL]: e.g. 0.05.
- e. Time span [s]: e.g. 120.

The titration is terminated when not more than 0.05 mL titrant is consumed within a time interval of 120 s. This condition can be effective at the earliest after 600 s. After 1800 s the titration is terminated even if the condition has not been met.

11. Enter the time limits for the evaluation:

- a. Time limit t1 [s]: e.g. 300
- b. Time limit t2 [s]: e.g. 1200

Note: The time limits are not restricted to the values t1 and t2 entered here. You can calculate, for instance in additional **Calculation** functions

- the titrant consumption between different time limits with R = QSTAT (100, 200).
- the titrant consumption up to a different time limit with R = VT (200).
- the correlation coefficient between different time limits with R = CSTAT (300, 600).

In this manner after a pH-stating you can use **Calculations** in the Analysis menu to define new time limits for results (see Section 3.12).

12. Enter the time interval [s] for the data storage: e.g. 10.

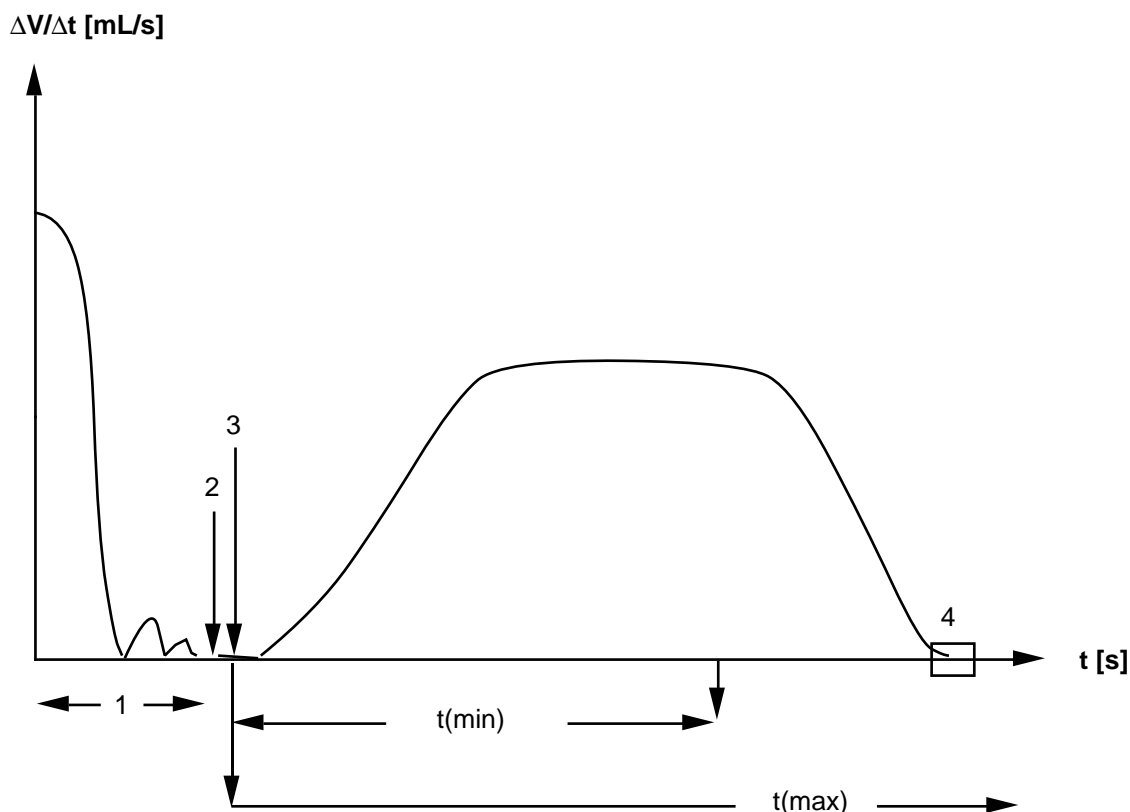
The current measured value and the associated volume are stored every 10 s. The titrator can store and print out maximum 300 measured values.

If you have entered an interval that is too small, the titrator first eliminates the values of the pretitration after 300 measured values. If the limit of 300 measured values is exceeded again, the titrator shortens the number by half after 300 measured values by leaving every second measured value in the memory. At the same time it automatically doubles the time interval.

13. Select a condition:

- No.
- Yes: Enter the condition.

Example of pH-stating profile



- 1: Pretitration
- 2: When the end point is attained, the titrator waits for 5 s before outputting an audio signal and sending the message "Pretitration complete: Please add sample". In the meantime, the titrator continues to stir and exert control.
- 3: Enter your sample and confirm the message with **RUN**: pH-stating begins.
Note: If you have to take away the titration vessel to add the sample, you can interrupt the pH-stating (see Section 3.4).
- 4: The termination condition $\Delta V/\Delta t$ has been met, the pH-stating is aborted.

Notes: a. The table of measured values of the record contains data of the pretitration and the pH-stating. If both titration steps call for more than 300 measured values the titrator stores only the measured values of the pH-stating.

- b. If you require a record of the curves, you receive only that of the actual pH-stating. You can follow the graphical representation of the pretitration only on the display.

2.3.14 Calculation

With the aid of this function you can calculate **one** result **R** for each sample.

Parameters of the mask:	Result name
	Formula
	Constant
	Result unit
	Decimal places
	Condition

1. Enter the result name: e.g. NaOH.
2. Select the formula from the recommendation menu or enter the one you need for your calculation.
3. Select the constant from the recommendation menu or enter the one you need for your calculation.
4. Select the result unit from the recommendation menu or enter the one you need for your calculation.
5. Enter the number of places after the decimal point that you require in the result.
6. Select a condition:
 - No.
 - Yes: Enter the condition.

Notes: a. You have a completely free selection of parameters 2-4 and you can interlink all available parameters and numeric values (see Sections 8.6.1 and 8.6.2).

b. The following operations are available for calculations:

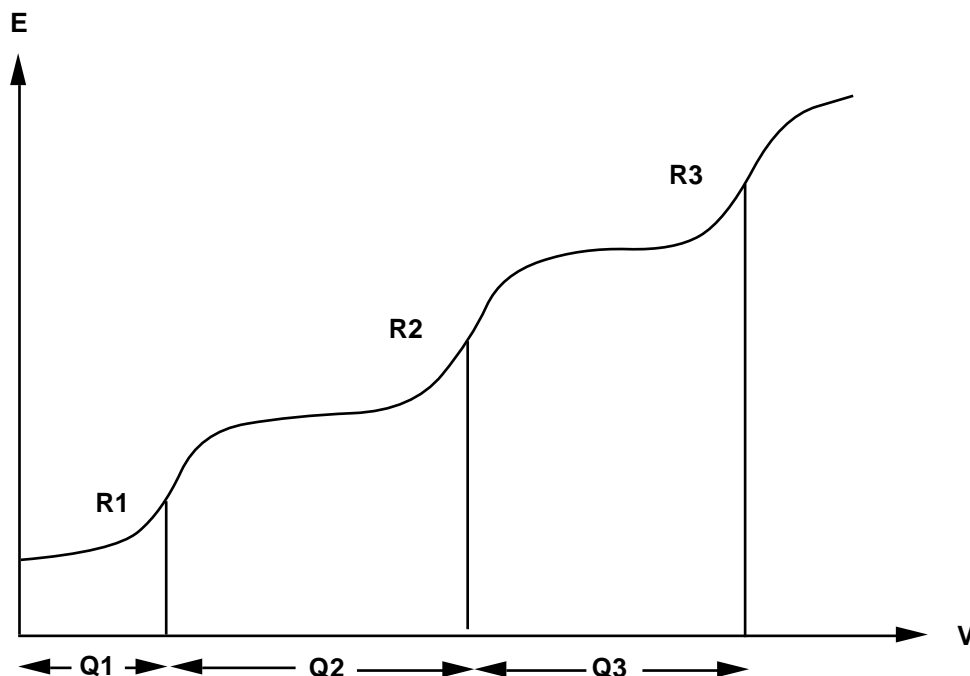
- | | |
|-------------------------------|-------|
| • Addition: | + |
| • Subtraction: | – |
| • Multiplication: | * |
| • Division: | / |
| • Logarithm to the base 10: | lg(x) |
| • Logarithm to the base e: | ln(x) |
| • Exponential to the base 10: | pw(x) |
| • Exponential to the base e: | ex(x) |
| • Square: | sq(x) |
| • Square root: | sr(x) |

- Notes
- c. If you press **HELP** you are shown for
Formula the most common formulae listed with the possible units and for
Constant the most common constants with the corresponding units.
 - d. You can have the formula as well as the constant stored as an auxiliary value and enter here as Hj: e.g. $R(R_i) = H(H_j)$ or $C(C_i) = H(H_j)$ (see *Auxiliary values*, Section 1.6 and *Use of indexes*, Section 8.2).
 - e. The titrator checks your entries when you quit the parameter mask with **EXIT**.
 If you have entered a wrong formula or constant, you will immediately be sent an error message (see Section 2.2.5: *Modify functions*, Note b.).
 If you have defined raw results (such as $R = QDISP$) which the titrator can not know, as a **Dispense** function did not precede this **Calculation** function, you will not be sent an error message until you try to save the method.

Example A: If you require, for instance, from an NaOH solution that you titrate with HCl not only the wt% NaOH as the result, but also the mmol consumption of HCl and the potential value of the equivalence point, you must specify three calculation functions for this method (see Section 8.1: *List of designations*, Section 8.2: *Use of indexes*, and method M001 in the Tutorial).

	<u>Formula</u>	<u>Constant</u>	<u>Unit</u>
1st Calculation function:	$R1 = Q$	Delete C with CE it is not needed	[mmol]
2nd Calculation function:	$R2 = EPOT$	Delete C with CE it is not needed	[mV]
3rd Calculation function :	$R3 = Q * C/m$	for $C = M/(10 * z)$	[%]

Example B: For the calculation of a titration curve with 3 equivalence points (acidic mixture of 3 substances) you also have to define the parameters of three **Calculation** functions. The respective constants C1, C2 and C3 are then also needed (see *Use of indexes*, Section 8.2).



	<u>Formula</u>	<u>Constant</u>	<u>Unit</u>
1st Calculation function:	$R1 = Q1 * C1/m$	for $C1 = M/(10 * z)$	[%]
2nd Calculation function:	$R2 = Q2 * C2/m$	for $C2 = (60.01 * 1000)/1$	[ppm]
3rd Calculation function:	$R3 = Q3 * C3/m$	for $C3 = 53.5/1$	[mg/g]

Notes: a. You must enter the molar mass M and the equivalent number z of the 2nd and 3rd **Calculation** functions as numeric values or insert as Hj if you have stored these as auxiliary values (see Section 1.6). **M** and **z** are defined in the **Sample** function only for the calculation of the first equivalence point (see Section 2.3.2).

b. If you do not know the number of the equivalence points, instead of defining indexes for Q you can have Q identified by a condition, e.g.

Q (200 < EPOT < 300) means that the Q used for the calculation is that whose equivalence point potential lies between 200 and 300 mV. If the titrator finds 2 equivalence points in this region it calculates the first one (see also Section 8.6.4).

- Notes: c. The titrator stores results until you start a new titration method or switch off the titrator.
- d. If you terminate a titration with **RESET**, all data recorded up to this point are evaluated. **Non-calculable** results (**R**) are then set to **zero** (0). If you were to abort the above titration shortly before the attainment of the second equivalence point, for instance, you would receive the result R1, but not R2 and R3.

2.3.15 Auxiliary value

Auxiliary value is an assignment function. The result (R or R_i) or its calculated mean value (\bar{x} or $\bar{x}[i]$) or a raw result of the titration method is assigned to the auxiliary value H_j and entered automatically with the date in the auxiliary value memory (see Section 1.6 and *Use of Indexes*, Section 8.2).

20 auxiliary value memories are available.

As an auxiliary value you can store, for instance:

- the blank value of a titration with the formula: " $H5 = \bar{x}$ " (example)
- a raw result, determined under the *Dispense*, *Measure*, *Temperature*, *Titration* and *pH/mV-stat* functions with the formula: " $H6 = VEQ$ " (example).

You can call up these auxiliary values for the functions

- **Titration** (value for EPS, values for buffer potentials P1 and P2),
- **pH/mV-stat** (value for EPS), and
- **Calculation**.

1. Enter an identification text: e.g. "Blank of DMF".

2. Enter the formula: " $H(H_j) = \bar{x}(\bar{x}[i])$ ", if you determine the auxiliary value with more than one sample, $n > 1$ and in this case the calculated mean value is assigned to the *Auxiliary value*.

Enter the formula: " $H(H_j) = R(R_i)$ " only if you determine the auxiliary value with one sample: $n = 1$.

Enter the formula: " $H(H_j) = E$ " only if a **Measure** function precedes the **Auxiliary value** function.

As a formula you can also enter, for instance: " $H(H_j) = \bar{x}[i] + QDISP$ " or " $H(H_j) = VTOT * 1.5$ ".

3. Select a condition:

- No.
- Yes: Enter the condition.

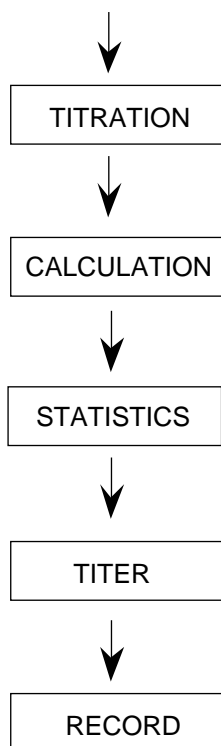
2.3.16 Titer

Titer is an assignment function: The result (R or R_i) or its calculated mean value (\bar{x} or $\bar{x}[i]$) of the titration of a titrant is assigned to the titer by the formula **$t = R1$ or $t = \bar{x}[i]$** and entered automatically with the date in the appropriate location of the titrant memory (see Section 1.1.2 and *Use of indexes*, Section 8.2).

1. Select the titrant to which you wish to assign the titer from the recommendation menu or enter one you have installed.
2. Enter its concentration [mol/L].
3. Enter the formula $t =$

\bar{x} (" $\bar{x}[i]$ ") if you determine the titer with more than one sample, **$n > 1$** and in this case the calculated mean value is assigned to the titer.
 Enter the formula $t =$

R (" R_i ") only when you determine the titer with one sample: **$n = 1$** .
4. Select a condition:
 - No.
 - Yes: Enter the condition.



Example of a method for the titer determination of a titrant with a primary standard: $n = 3$.

Calculation

You define the following result: R (or R_i) = $m / (VEQ * c * C)$;
 $C = M / (1000 * z)$

Statistics

The mean value of the result R (or R_i) is calculated for the 3 samples (see *Statistics* function, Section 2.3.18).

Titer

The calculated mean value is assigned to the titer by the formula **$t = \bar{x}$ or $(\bar{x}[i])$** .

2.3.17 Calibration

You use this function to calibrate an electrode: its zero point and its slope are calculated. You determine the buffers to be used for the calibration of the sensor. The potential of the buffer solutions is determined with the aid of the **Measure** function (see example at the end of the section).

Notes: a. Since the slope of an electrode is temperature dependent, it is important to specify the temperature at which the calibration is performed.

- Before the start of the **Measure** function, the temperature of the buffer solution is determined automatically if a temperature sensor is attached. Otherwise the temperature entered at the start of the calibration method will be adopted (see *Sample* function, Section 2.3.2).

If you measure the pH value of a solution at a different temperature at a later date, the slope of the sensor is temperature compensated by the titrator.

- b. The calibration data (zero point, slope and calibration temperature) are transferred automatically with the date to the installation data of the sensor (see Section 1.2.2).
- c. Depending on the number of buffers measured, the titrator performs the following calibration:
 - With one buffer sample ($n = 1$) it calculates the zero point of the sensor. The slope remains unchanged.
 - With several samples ($n > 1$) it calculates the zero point and the slope of the sensor.

1. Select the sensor from the recommendation menu or enter the one you have installed (see Section 1.2).

2. Select the buffer type from the selection menu:

For DIN/NIST, MERCK Titrisol and INGOLD buffers the titrator has 8 values stored in each case and you can select these to calibrate pH electrodes. You can enter the values of buffer solutions you have chosen yourself under "pH, pM, pX (free selection)" to calibrate pH or ion-selective electrodes.

- a. DIN/NIST buffer [pH]: The buffer values apply to a temperature of 25 °C.

- Select the buffer value for the 1st buffer from the selection menu, e.g. pH 1.679.
- Select the buffer value for the 2nd buffer from the selection menu, e.g. pH 6.865
- etc..

- b. MERCK Titrisol buffer [pH]: The buffer values apply to a temperature of 20 °C.
 - Select the buffer value for the 1st buffer from the selection menu, e.g. pH 4.
 - Select buffer value 2 for the 2nd buffer from the selection menu, e.g. pH 7.
 - etc..
 - c. INGOLD buffer [pH]: The buffer values apply to a temperature of 25 °C.
 - Select the buffer value for the 1st buffer from the selection menu, e.g. pH 4.6.
 - Select the buffer value for the 2nd buffer from the selection menu, e.g. pH 9.21.
 - etc..
 - d. pH, pM, pX buffers. You have a free choice of maximum eight buffer values.
 - Enter the value for the 1st buffer.
 - Enter the value for the 2nd buffer.
 - etc..
3. Enter Ri (i = index): see *Use of indexes*, Section 8.2.
 4. Enter the minimum slope [mV/unit] , e.g. 53.
 5. Enter the maximum slope [mV/unit] , e.g. 61.

Notes: a. The stored buffer values, types a, b. and c, apply to the specified temperature. If you calibrate at another temperature, these buffer values will be temperature-corrected automatically and recorded on the printout.

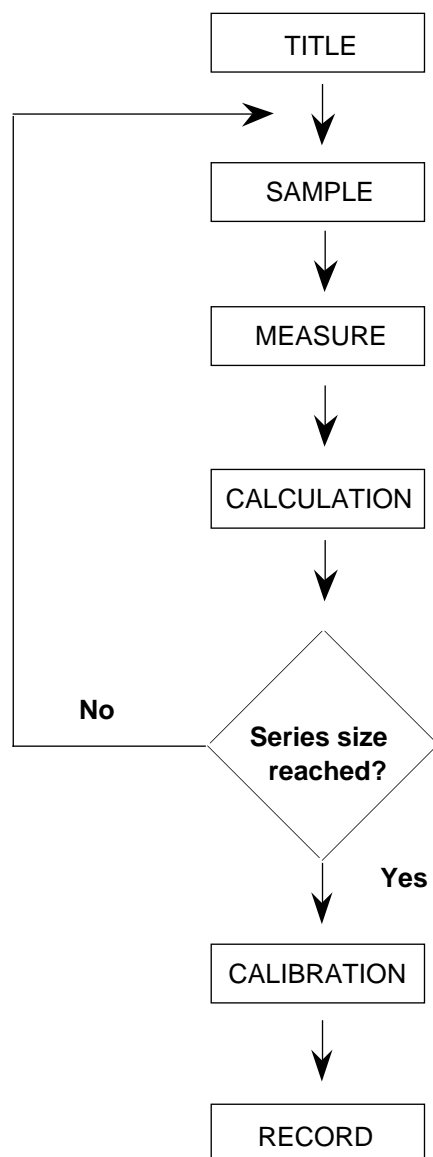
- b. You have specified 3 MERCK Titrisol buffers in a calibration method:
 - first buffer: pH 4,
 - second buffer: pH 7,
 - third buffer: pH 9.

When you measure these 3 solutions the order is immaterial. If you measure only 2 solutions, however, you must measure the first and second buffer (pH 4 and pH 7) in that order otherwise you will obtain wrong calibration data!

If you measure with buffer solutions whose values you have entered under point 2d, you must keep to the order defined there!

- c. If, in a pH calibration, with e.g. these 3 buffer types you obtain a slope that exceeds the limits you defined, the calibration data of the appropriate sensor are **not** entered. The error message "Data not acquired" appears on the record.

Example of a calibration method

**Sample**

You define only the number of samples, that is the number of buffers which you use to calibrate the electrode, e.g. $n = 3$. You can leave all other parameters as they are, the titrator ignores them. As soon as it has executed this function, it begins to stir at the default speed.

Measure

The titrator acquires the measured value E in each case.

Calculation

You define a result: $R = E$. This stores the 3 measured values of the 3 buffer solutions.

Calibration

The titrator assigns the acquired measured values of the **Measure** function to the values of the 3 buffer solutions and in this manner calculates the zero point and the slope of the electrode.

The entry R_i ($i = \text{index}$) must be the same as in the **Calculation** function (in this case it is R).

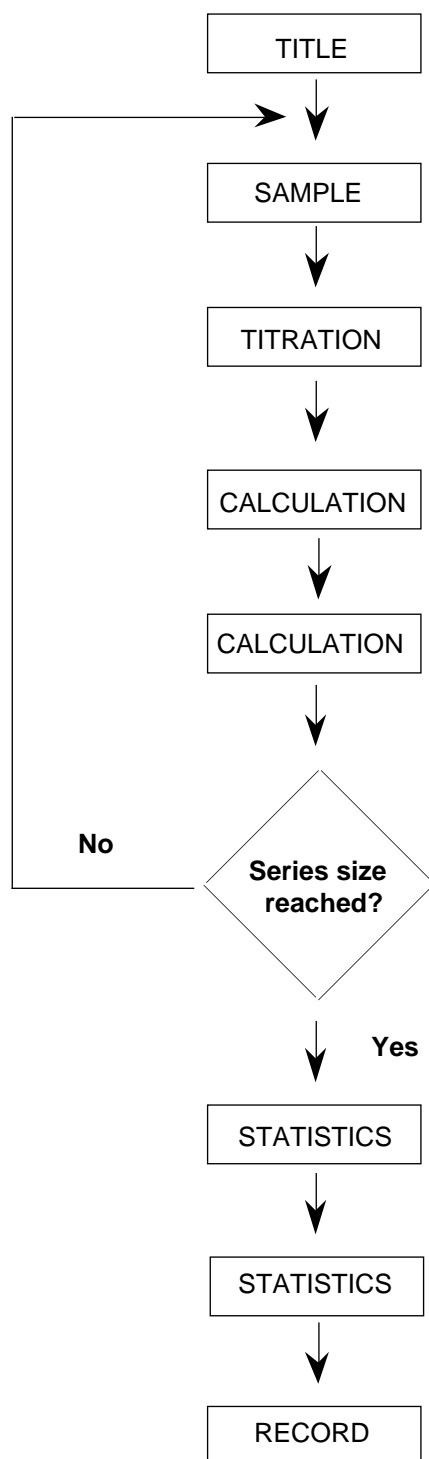
2.3.18 Statistics

With the aid of this function you obtain a statistical evaluation of the results of a series titration. The titrator calculates the mean value \bar{x} automatically when you have the **Statistics** function in your method. If you nevertheless titrate only one sample with this method, the titrator ignores the function.

1. Enter R_i (i = index): see *Use of Indexes*, Section 8.2 and the following chart.
2. Select the standard deviation **s**:
 - "Yes" or "No".
3. Select the relative standard deviation **srel**:
 - "Yes" or "No".
4. Select outlier test
 - No.
 - Yes: The titrator checks the results in accordance with an outlier test following Grubbs. It removes outliers and repeats the statistical evaluation.

The outlier test is not performed until the number of samples n is greater than 3: $n > 3$.

Example of a titration method for a known acid mixture (2 equivalence points) where the percent content of both components with corresponding standard deviation is required as result:



Sample

The number of samples is **n = 5**.

The titrator begins to stir at the default speed as soon as it has executed this function.

Titration

The titrator acquires the raw results Q1 and Q2.

Calculation

You define the result **R** (R1) for the evaluation of the first equivalence point: $R = Q1 * C1/m$ (see *Calculation* function, Section 2.3.14).

Calculation

You define the result **R2** for the evaluation of the second equivalence point: $R2 = Q2 * C2/m$.

Statistics

The entry Ri (i = index) must be the same as in the **Calculation** function:

In the first **Statistics** function, **R**,

in the second **Statistics** function, **R2**.

The titrator assigns the 5 results **R** or **R2** of the 5 samples to the statistics functions and thus calculates the mean values \bar{x} and the corresponding standard deviations.

2.3.19 Record

With the aid of this function you determine what data should be recorded where.

Notes: a. If in your method a **Record** function **follows** the **Statistics** function the titrator records only the selected parameters of the **last** sample of a titration. If you add a **Record** function **before** the **Statistics** function, the titrator records the selected parameters of all samples (see following).

- b. If your method has two or more **Titration** functions and you would like to have a record of the table of measured values and curve of the first (second or third) titration, a **Record** function must follow the **Titration** function as the titrator stores only the measured values of the last **Titration** function (see Examples of methods, Section 8.7.2).

The titrator stores the raw results of all functions up to the titration of the next sample within a loop (see Section 8.5.7).

1. Output unit

In the selection menu choose between:

Printer

Computer

Printer + Computer

Note: **Printer** is stored as the default parameter (the printer you have installed, see Section 1.8.1).

If you have not installed a printer and/or computer, the function can not be executed. However, you can leave it in the method since in this case it neither triggers an error message nor influences the titration.

If you have installed a printer and/or computer, but have not connected it or switched it on,

- the titrator waits until you switch on the printer and starts to transfer its data,
- the computer sends an appropriate error message.

2. Short form method

Would you like a record of the method in short form?
Select "Yes" with **SEL**.

3. Sample data

Would you like a record of all sample data (ID1, ID2, molar mass, equivalent number, weight/volume, correction factor) of the completed titration method?
Select "Yes" with **SEL**.

-
- | | |
|--|--|
| 4. Raw results last sample | Would you like a record of all raw results such as VEQ or VDISP of the last sample?
Select "Yes" with SEL . |
| 5. Results last sample | Would you like a record of all results of the last sample?
Select "Yes" with SEL . |
| 6. All results | Would you like a record of all results of the completed titration method?
Select "Yes" with SEL . |
| 7. Table of measured values | Would you like a record of the table of measured values of the last sample?
Select "Yes" with SEL . |
| 8. E - V curve | Would you like a record of the titration curve potential vs volume of the last sample?
Select "Yes" with SEL . |
| 9. $\Delta E/\Delta V$ - V curve | Would you like a record of the 1st derivative of the titration curve potential vs volume of the last sample?
– (The ordinate scale is linear.)
Select "Yes" with SEL . |
| 10. $\log \Delta E/\Delta V$ - V curve | Would you like a record of the 1st derivative of the titration curve potential vs volume of the last sample?
– (The ordinate scale is logarithmic.)
Select "Yes" with SEL . |
| 11. $\Delta^2 E/\Delta V^2$ - V curve | Would you like a record of the 2nd derivative of the titration curve potential vs volume of the last sample?
– (The ordinate scale is linear.)
Select "Yes" with SEL . |
| 12. E - t curve | Would you like a record of the titration curve potential vs time of the last sample?
Select "Yes" with SEL . |
| 13. V - t curve | Would you like a record of the titration curve volume vs time of the last sample?
Select "Yes" with SEL . |
| 14. $\Delta V/\Delta t$ - t curve | Would you like a record of the titration curve of the 1st derivative volume vs time of the last sample?
Select "Yes" with SEL . |
-

15. Select a condition:

- No.
- Yes: you are shown the following mask:

Condition

Termination after record

If you select "Condition", you must enter a condition.

If you select "Termination after record", you have two possibilities:

- "No" or "Yes".

With this termination parameter you can abort a method when the defined condition is satisfied.

Example

In your method the number of samples is $n = 3$.

You wish to abort the method when the titrator finds no equivalence point in one of the samples but would like to use a recorded table of measured values or titration curve for possible determination of the reason for this.

The condition in this case is $neq = 0$ (see Section 8.1: *List of designations* and Section 8.3: *Functions with a condition*).

Your method should comprise the following functions for this:

Title

Sample

Stir

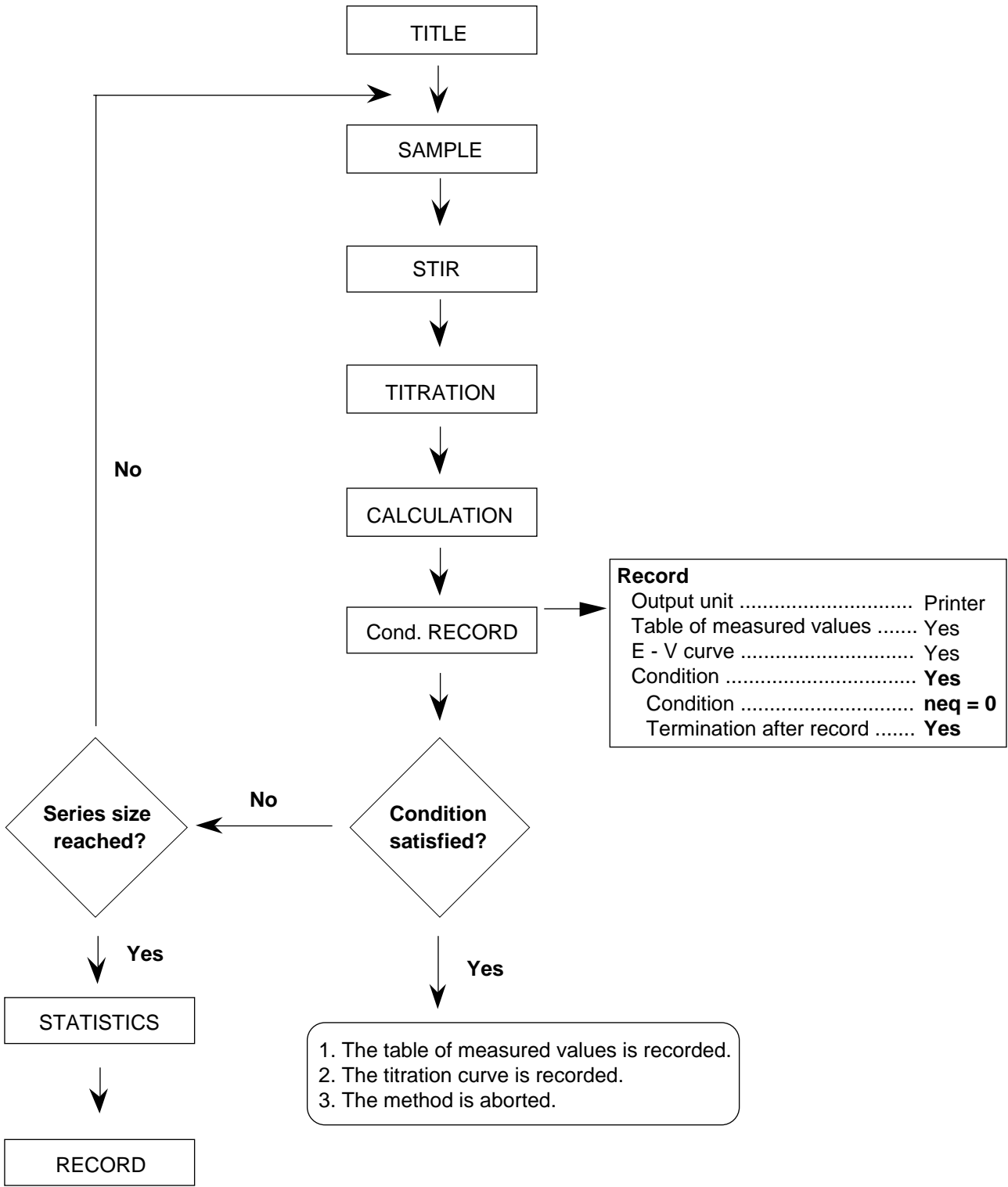
Titration

Calculation

Record (function with condition: table of measured values and titration curve are selected.)

Statistics

Record



2.3.20 Sync

Under this function you specify the parameters for the synchronization of the titrator with an external control unit. The synchronization takes place via the system interface.

1. Select the synchronization mode:
 - "Send" or "Send/Wait".
 - a. In the synchronization mode **Send** the titrator transmits the numeric code you have defined to the external control unit and then begins to process the next method function immediately.
 - b. In the synchronization mode **Send/Wait** the titrator transmits the numeric code you have defined to the external control unit and then waits until this sends back the same code. Only then does the titrator start to process the next method function.
2. Enter the code: A number between 1 and 32!
3. Enter a comment in the space foreseen for it.
This comment appears on the display when the function is executed.
4. Select a condition:
 - No.
 - Yes: Enter the condition.

- Notes:
- a. You will find additional information regarding the communication between the titrator and the external control unit in Section 7.
 - b. Two titrators can also be synchronized by means of the system interface using the SYNC function (see Operating Instructions "RS232C Interface Description" provided with the RS option).
 - c. With the DL77, it is possible to use the SYNC function to synchronize the two method lists in Analysis menus A and B (see Operating Instructions "RS232C Interface Description" provided with the RS option).

Contents	Page
3. ANALYSIS	3-3
3.1 Executing a method	3-4
3.1.1 Notes on weight entry	3-9
3.1.2 Notes on method and sample data mask	3-10
3.1.3 Titration sequence	3-12
3.1.4 Restarting a previously executed method	3-14
3.2 Terminate current method (RESET)	3-15
3.3 Fade out current method	3-16
3.4 Interrupt current method	3-17
3.5 Reevaluation	3-19
3.6 Modify current method	3-21
3.7 Sample data	3-23
3.8 Method data	3-26
3.8.1 Record data	3-27
3.9 Display	3-28
3.9.1 Representation	3-29
3.9.2 Curve type	3-30
3.10 Stirrer	3-31
3.11 Records	3-32
3.12 Calculations	3-34

	Page
3.13 Method list	3-35
3.13.1 Filling the method list	3-35
3.13.2 Modifying the method list	3-36
3.13.2.1 Cut	3-36
3.13.2.2 Paste	3-36
3.13.3 Processing the method list	3-37
3.13.3.1 Single method	3-37
3.13.3.2 List once	3-37
3.13.3.3 List continuous	3-40
3.14 Parallel titrations with the DL77	3-42
3.14.1 Notes on parallel titrations	3-43
3.14.2 Changing the Analysis menus	3-44
3.15 Sample series with 2 sample changers	3-45

3. ANALYSIS

Under this menu you perform the titration with a selected method. The necessary data are stored as a method in the EDITOR and as resources in INSTALLATION.

You prepare your sample, titrate and receive the result. You have the possibility to effect entries during the titration or to interrupt it to modify parameter values of the method.

You can enter the methods you wish to run with the sample data weight or volume in advance; the titrator then calls up the methods in succession and performs the titrations with your help. An attached sample changer automates this operation.

After every sample determination you can perform additional calculations and print out measured values or curves that you have not specified under the corresponding method function.

This menu demonstrates the differing capabilities of the three titrators:

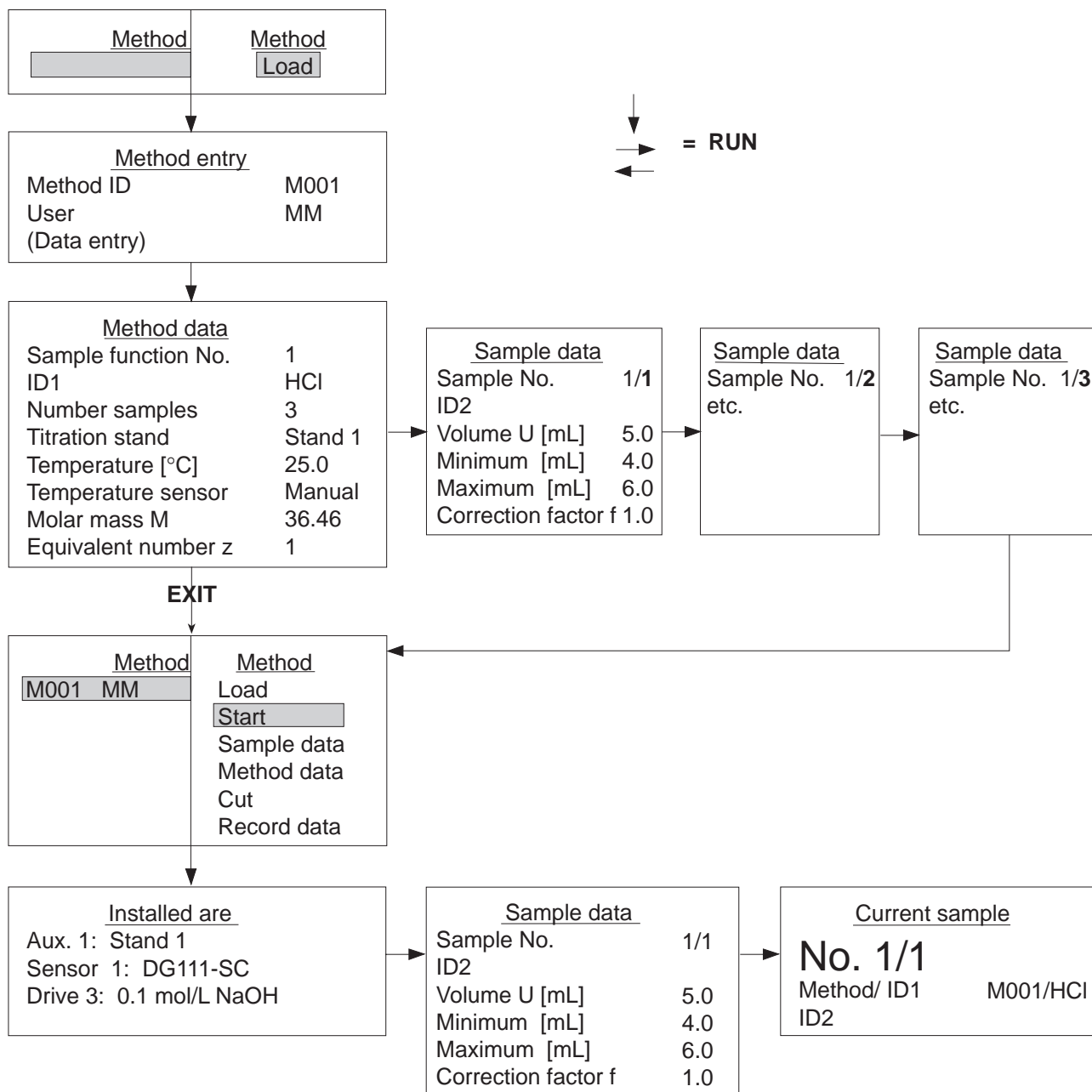
DL67: Only **one** method can be processed at a time.

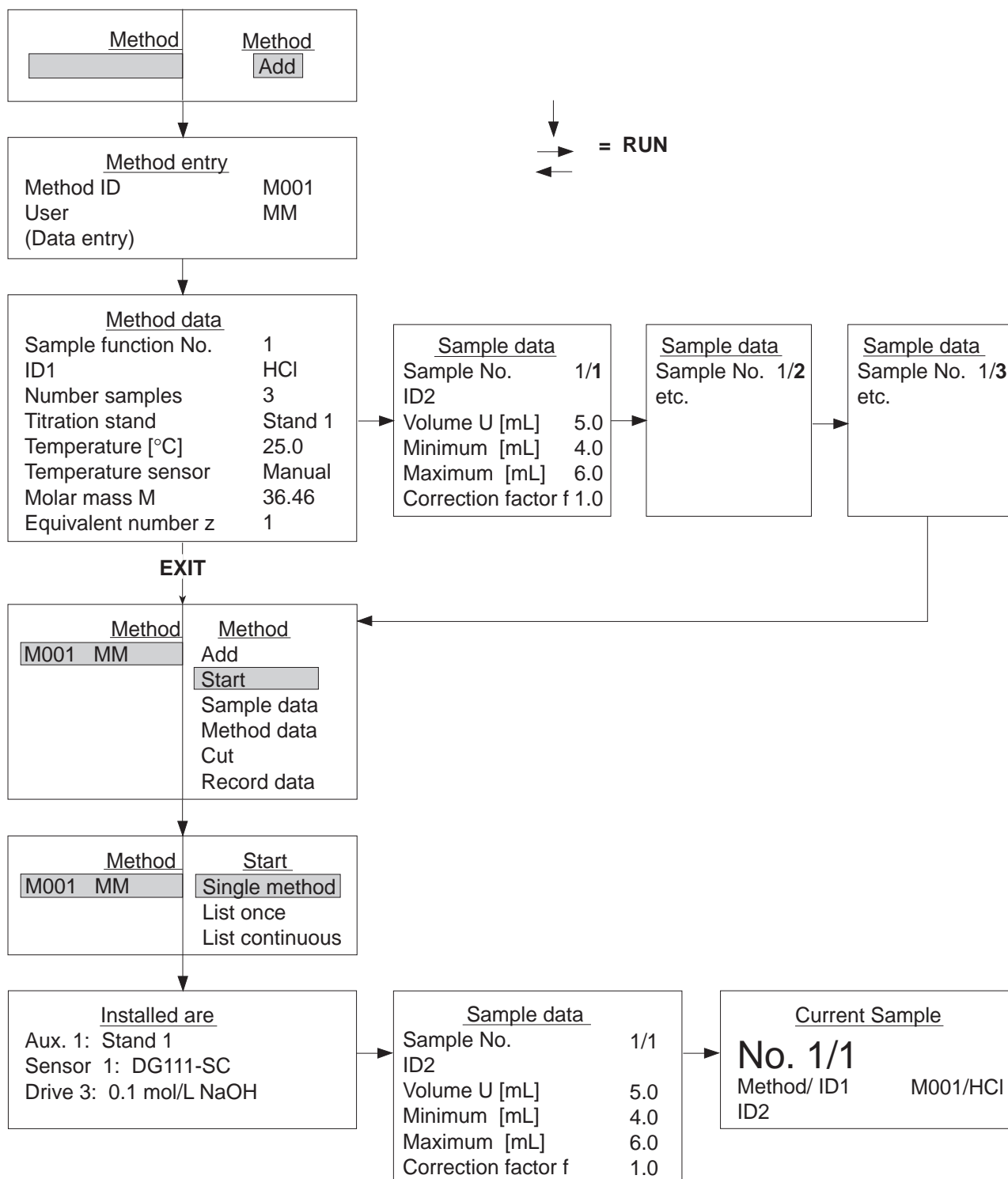
DL70ES: Up to **10** methods can be entered in a list and run automatically in succession.

DL77: **10** methods can be entered in **each** of **two** lists and run in **parallel**.

3.1 Executing a method

DL67: Menu sequence until the start of a method - with **RUN** and/or **EXIT**



DL77/DL70ES: Menu sequence until the start of a method - with **RUN** and/or **EXIT**

DL77: On the right side of each mask (possibly in the side bar) the letter **A** or **B** indicates the selected Analysis menu.

When you select **ANALYSIS**, the method list appears together with the selection menu (see diagram of the sequence on the previous pages). The list is either

- blank, indicating that you must **add** a method, or
- it contains one or several methods already, indicating that you can **start** one of them (see Section 3.13).

DL67: Since you can store only one method in the method list, you **load** this method (see page 3-8: *Caution DL67*).

– Confirm **Add (Load)** with **RUN**: The method entry mask appears.

Method ID	Enter the identification ID of the desired method.
	If no method with the specified identification is stored in the titrator and you have installed a computer, the method will be requested from the computer and added to the method list (see Operating Instructions provided with the RS option).
User	Enter your name and press RUN .
(Data entry)	appears only, if you have installed a computer. For further details about the communication between the titrator and computer, see Section 7.1 and the Operating Instructions provided with the RS option.

You are shown the method data mask with the following parameters:
(example: determination of CaCl_2 , see Sections 3.1.2 and 3.1.3)

<u>Method data</u>	
Sample function No.	1
ID1	Ca457
Number samples	3
Titration stand	Stand 1
Temperature [°C]	25.0
Temperature sensor	Manual
Molar mass M	110.99
Equivalent number z	2

1. **1** refers to the **first Sample** function within the method. You can not change this specification (see also Section 3.1.2).
2. You entered identification 1, which applies to all samples of this method, under the **Sample** function. Here, you can modify or delete this entry (see Section 2.3.2).
3. You can modify the number of samples entered under the **Sample** function ($n = 1$ to 60).

4. You can modify the titration stand entered under the **Sample** function (see Section 1.7).
5. Enter the current temperature of the solutions to be titrated, if you have not attached a temperature sensor (*Temperature sensor: Manual*).
6. You can modify the temperature sensor entered under the **Sample** function.
7. You can modify the molar mass M entered under the **Sample** function, for example if you have to determine MgCl_2 instead of CaCl_2 .
8. You can modify the equivalent number entered under the **Sample** function.

If you confirm one of the parameters of the method data mask with **RUN**, you are shown the sample data mask (see also Section 3.1.2):

<u>Sample data</u>	
Sample No.	1/1
ID2	
Weight m [g]	0.0
Minimum [g]	0.1
Maximum [g]	0.2
Correction factor f	1.0

1. **1/1** refers to the **first Sample function** and the **first** sample and can not be changed (see Section 3.1.2).
2. Enter an identification for **this** sample.
3. Enter the weight m [g] within the limits specified in the **Sample** function or have it transferred by an attached balance (see Section 3.1.1).
(Sample preparation: weigh in CaCl_2 and add 40 mL deion. H_2O).
4. You can not change the minimum and maximum values specified in the **Sample** function.
5. For every sample you can enter a value which will be calculated, if you incorporate **f** in the formula of the **Calculation** function.

Example: Each sample has a different, known moisture content, which can be compensated by incorporation of factor **f**.

1st sample: moisture content = 4% → **f = 0.96** → $R = Q * C / (m * f)$

2nd sample: moisture content = 3% → **f = 0.97** → $R = Q * C / (m * f)$

Once you have confirmed the entries with **RUN**, you will be shown the sample data mask for the second, then the third sample, in order to enter the weight, before the following selection menu appears:

DL77/DL70ES	
	see Section
<u>Method</u>	
Add	3.1,3.13.1
Start	3.1,3.1.4,3.13.3
Sample data	3.7
Method data	3.8
Cut	3.12.2.1
Record data	3.8.1

DL67	
	see Section
<u>Method</u>	
Load	3.1,3.13.1
Start	3.1.4
Sample data	3.7
Method data	3.8
Cut	3.12.2.1
Record data	3.8.1

You can select all menus or execute all commands of this selection menu before starting the method.

Caution DL67: If you now confirm **Load**, the titrator will automatically load the same method. Modified method data and entered sample data are deleted! The command allows a "new" method to be loaded if you modify the identification of the old one.

- Confirm **Start** with **RUN**.

The following selection menu appears (**not** on the DL67, see page 3-4: *Menu sequence*):

Start
Single method
List once
List continuous

- Confirm **Single method** with **RUN**.

You are shown the installed resources, to check if your installation matches this information.

- Notes:
- If you have not installed any resources, you are shown the error message "Installation data missing for" (e.g. 1 mol/L AgNO₃).
 - If you have excluded the appearance of "Installation data" in **Analysis parameters**, the mask "Installed are" will not appear (see Section 1.9.8).
 - Up to this mask, **EXIT** or a key combination (index + letter) can be used to leave the menu sequence before the start of the actual titration. Values entered up to this point are saved.

If you confirm the installation data with **RUN**, the titrator will process the method.

3.1.1 Notes on weight entry

- a. If you violate the upper or lower weight limits, but nevertheless confirm this entry with **RUN**, you are shown the following message:

Entry outside limits

Modify entry

Save entry

If you confirm "Modify entry" with **RUN** you are again shown the sample data mask to allow you to change the weighing.

If you confirm "Save entry" with **RUN**, the titrator follows the menu sequence.

- b. If you have the weight transferred by a balance, press **SEL**. You are shown the mask

Weight

0.00000 → (0.13460)

Accept weight

– When the weight value is stable, confirm "Accept weight".

If the weight is below the minimum, three minus signs (- - -) appear after "Weight", if it is above the maximum three plus signs (+ + +) are displayed. You can modify the weighing or confirm it (see Note a).

- c. If you have selected the **bidirectional** transmission mode, you can accelerate the entry process:

– Press **SEL** at the weight entry "0.0000" in the first sample data mask.

The sample number, e.g. **no. 1.1** is faded in on AT, AM, and PM balances.

– Press the TARA key, so that the current weight appears on the display of the balance. With these balances the weight limits also appear so that you can change the weight if need be.

– Confirm the weight transfer with the **Print** key of the balance: the titrator automatically readies itself for the weight entry of the next sample.

In this manner you can enter the weight values of all samples of a method without pressing a key of the titrator.

- d. If you have the "Mettler DataPac-M" connected to AM or PM balances, you can enter the ID2 on this.

– Press <CLEAR>,

– enter ID2,

– press <ENTER>.

3.1.2 Notes on method and sample data mask

- a. If there is no **Sample** function in a method, you are shown neither the method data mask, the sample data mask nor the request regarding the current sample (see **Sample** function, Section 2.3.2).
- b. If the method has **two Sample** functions the method data mask reappears after confirmation with **RUN**. In this case a **2** follows **Sample function No.**
- c. If you have selected "Fixed volume" in the Sample function and have specified the volume, only ID2 and the correction factor can be entered or modified in the sample data mask. The entry limits "Minimum" and "Maximum" are missing. If you do not need these entries, you can skip this mask by quitting the previous method data mask with **EXIT**.
- d. If you confirm the sample data mask with **RUN**, a mask for each sample of the designated number of samples in this **Sample** function will be shown in succession (for several **Sample** functions, masks for the other function's samples will be shown as well: **Sample No. 2/1** and so on).

Sample data need not be entered at this point. You can circumvent the sample data masks by leaving the previous method data mask with **EXIT** (see menu sequences on pages 3-4, 3-5 und 3-11).

- The sample data mask will then appear prior to the request "Current sample". You can enter the weight/volume for the first sample only.
- The mask will not reappear if you have specified results within a method under the **Calculation** function that do not need the weight/volume entry, in other words the titrator needs no entries.

Example: $R = VEQ$ or $R = Q$

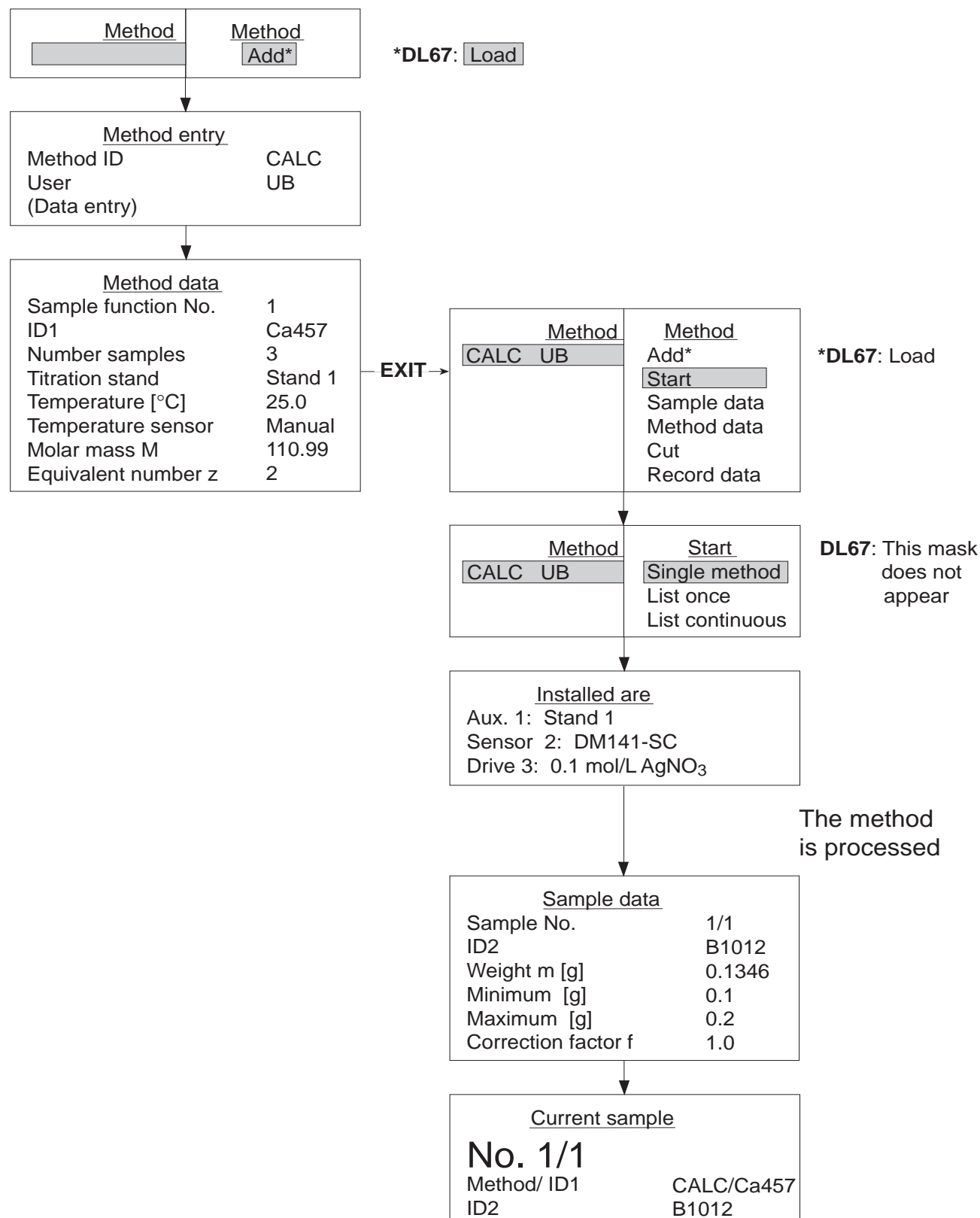
- e. If you must enter the weight or volume during the titration, for example, when no time may be lost by back-weighing,
 - confirm the sample data mask with **RUN** without entering the weight.

Since the titrator does not need the sample data until it performs calculations, the sample data mask reappears in the display, either

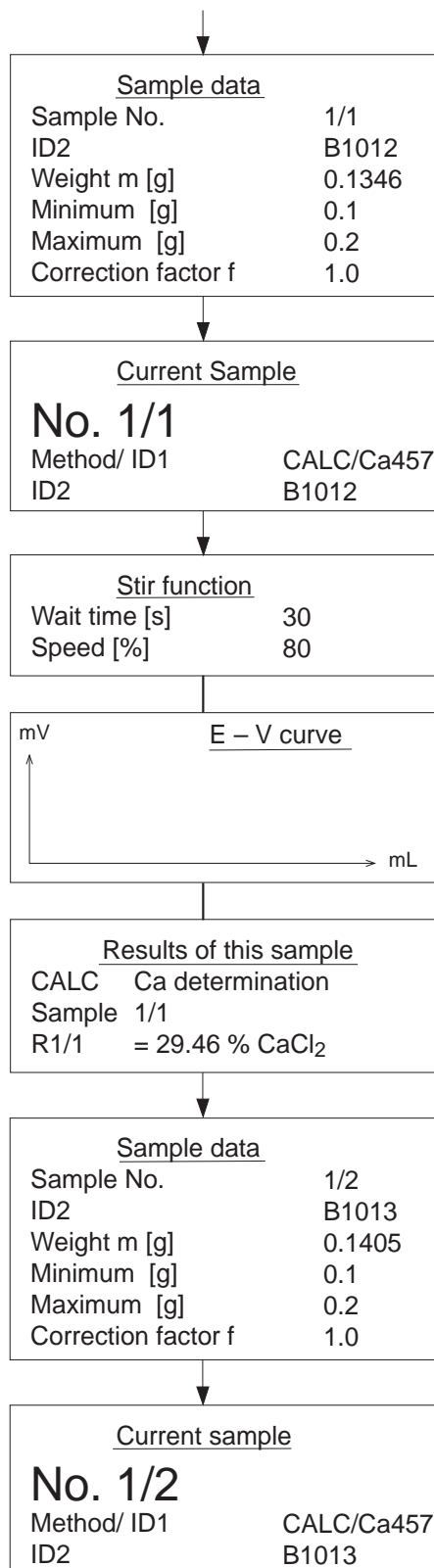
- when the titrator reaches the **Titration** function in which a predispensing to nominal content or a termination criterion after nominal content has been specified or
- when it reaches the **Calculation** function in which the weight **m** is needed for the result (see *Examples of methods*, Section 8.7.1).

You also have the possibility to fade out the titration with **EXIT** and enter the weight in the selection menu *Sample data* (see Sections 3.3 and 3.7).

Example of the menu sequence when weight volume must be entered during the active method:



3.1.3 Titration sequence (excerpt of the described method)



The titrator processes the method and has reached the **Sample** function:

- Enter ID2.
- Weigh in the sample and enter the weight for the **first** sample or have it transferred by a balance.
- Press **RUN**.

The titration vessel must now be attached to the titration stand.

The titrator stirs at 80% maximum speed and waits 30 s (CaCl₂ is dissolved during this time). The running time is shown.

The titrator processes the **Titration** function.

The first sample has been titrated. You are shown the result of this sample.

- If you have excluded "Results of this sample" from appearing using **Analysis parameters**, the titrator will skip this information (see Section 1.9.8).

With **RUN** the sample data mask for the **second** sample appears:

- Enter ID2.
- Weigh in the sample, enter the weight or have it transferred by a balance.
- Press **RUN**.

You are "requested" to insert the second sample, etc.

Notes: a. The titrator acquires the running time for each processed method as the raw result TIME (see *List of designations*, Section 8.1 and *Karl Fischer Titrations*, Section 10.2.7):

Start of time acquisition: see diagram page 3-11: "The method is processed".

End of time acquisition: The result list appears on the display (single method) provided that "Results of this sample" has been confirmed.

- b. If you have specified **Auto stand** as the titration stand in your method, the mask "Current sample" will not appear in the titration run. This means that the next sample determination will be started **without** a **RUN** confirmation. It is either not necessary to change the sample, or it will be changed by a robot driven by the **Sync** function.

At the end of a titration method you are shown the results for all samples on the display only when the titrator has transferred all data to the printer (only when the method has **Record** as its last function). During this operation the message "Output unit writing record" appears.

Note: All results of a learn titration are marked on the display with an exclamation mark (!).

If you confirm the result list with **RUN**, the selection menu appears:

DL77/DL70ES	
<u>Method</u>	see Section
Add	3.1/3.13.1
Start	3.1/3.1.4/3.1 3.3
Sample data	3.7
Display	3.9
Records	3.11
Calculations	3.12
Method data	3.8
Cut	3.13.2.1
Record data	3.8.1

DL67	
<u>Method</u>	see Section
Load	3.1/3.1.4
Start	3.1/3.1.4
Sample data	3.7
Display	3.9
Records	3.11
Calculations	3.12
Method data	3.8
Cut	3.13.2.1
Record data	3.8.1

You can select all menus or execute all commands of this selection menu before starting the next method.

Note: The executed method is marked by an asterisk (*).

Before you start the next method you can reexamine the sample data or have them printed out (see Sections 3.7 and 3.8.1).

As soon as you start the next method all data of the executed method are deleted.

3.1.4 Restarting a previously executed method

If you select the previously used method and confirm **Start**, the titrator reloads this method automatically. In doing this, stored modified method data are retained, whereas sample data entered for the previous series are deleted and must be reentered for the subsequent samples.

DL67: The titrator reloads a previously executed method if you confirm this method with **Load**. Modified method data and previously entered sample data are all deleted.

3.2 Terminate current method (RESET)

If you wish to terminate the method, press **RESET**. The titrator **interrupts** it immediately. The following mask appears (**RESET** was pressed during the second sample's **Titration** function):

Terminate

```
ANALYSIS A*; Method: CALC          ( * appears only with the DL77)
Sample No. 1/2
Titration [1]
```

If you simply wish to terminate the (first) **Titration** function,

- confirm "Titration [1]" with **RUN** and quit the mask with **EXIT**: The titrator terminates the **Titration** function definitively; the subsequent functions of this sample determination are, however, processed.

If you simply wish to terminate the determination of the current sample,

- confirm "Sample No. 1/2" with **RUN** and quit the mask with **EXIT**: The titrator terminates the determination of this sample definitively and "Results of this sample" appears on the display. You are asked for the third sample with **RUN**.

If you wish to terminate the **method**,

- confirm "ANALYSIS A; Method: CALC" with **RUN** and quit the mask with **EXIT**: The titrator terminates the method definitively and you are shown the results calculated up to this point on the display.

Notes: a. If data have been transferred to the connected output unit already, these are printed out/written.

- b. If the titrator is executing several activities (e.g. auxiliary functions), they are **interrupted** and displayed, too (see Section 4, Note c.).

3.3 Fade out current method

If you wish to enter sample data **during** a titration or wish to modify the stirrer speed or the curve type of the representation (*Titration* or *pH/mV-stat* function), press **EXIT**. The following selection menu appears:

DL77/DL70ES		DL67	
	see Section		see Section
<u>Method</u>		<u>Method</u>	
Add	3.1/3.13.1	Interrupt	3.4
Interrupt	3.4	Sample data	3.7
Sample data	3.7	Display	3.9
Display	3.9	Stirrer	3.10
Stirrer	3.10	Method data	3.8
Method data	3.8	Record data	3.8.1
Record data	3.8.1		

The titration continues to run in the background.

It also continues to run if you use the required key combination (index + letter) to select another menu.

- To fade in the titration again, press **<index + T>**:

If the titrator is still processing the **Titration** function, the curve or table of measured values, for example, appears on the display; if not the method function is displayed that the titrator is processing at the time.

DL67: Jumping to other menus are impossible during a titration. You can only select menus, i.e. the commands in the selection menu.

The following key combinations are possible:

- **<index + T>** (functions of the current method, see above)
- **<index + S>** (sample data list, see Section 3.7).

3.4 Interrupt current method

If you select **Interrupt** in the selection menu "Method", the titrator discontinues the function it is currently processing. The following selection menu appears:

DL77/DL70ES		DL67	
	see Section		see Section
<u>Method</u>		<u>Method</u>	
Add	3.1/3.13.1	Continue	see below
Continue	see below	Sample data	3.7
Sample data	3.7	Display	3.9
Display	3.9	Stirrer	3.10
Stirrer	3.10	Records	3.11
Records	3.11	Calculations	3.12
Calculations	3.12	Method data	3.8
Method data	3.8	Modify method ^{a.}	3.6
Modify method ^{a.}	3.6	Reevaluation ^{a., b.}	3.5
Reevaluation ^{a., b.}	3.5	Record data	3.8.1
Record data	3.8.1		

^{a.} If you interrupt a METTLER method, the menus "Modify method" and "Reevaluation" are missing. This also applies to methods stored only in the computer (see *Method ID*, Section 3.1).

^{b.} "Reevaluation" appears only when you interrupt a user method during a **Titration (EQP)** function for which a possible predisensing has been completed.

You can select or execute all menus or commands of the selection menu. To continue titration – confirm **Continue** with **RUN**.

The functions **Titration**, **pH/mV-stat**, **Dispense**, **Measure**, or **Temperature** are continued from the point at which they were interrupted!

Caution: The remaining functions are repeated!

Example: In the **Stir** function, the solution is stirred for the specified time, regardless of the time stirred before the interruption.

In most cases you interrupt the determination to modify parameter values of the current method (see Section 3.6).

The titrator itself interrupts a titration immediately in the following situations:

1. After the **Titration** mode **LEARN EQP** or **LEARN EP** if it has not found an equivalence or end point (see also Sections 2.3.12.4/5).

- Confirm the error message with **RUN**: The selection menu reappears (see previous page).

If you wish the titrator to perform the remaining functions of the method,

- select **Continue**: The titration resumes and the representation shown before the interruption reappears on the display.

If you wish to terminate the method,

- press **RESET** (see Section 3.2).

2. During a **Titration** function if the condition for the specified parameter **Stop for reevaluation** is satisfied (see next section).

3.5 Reevaluation

You can reevaluate the titration curve in an equivalence point determination (EQP) by modifying the titration parameters. This is possible only while the **Titration** function is active. Either

- you interrupt the current **Titration** function after a possible predisensing has been completed (the selection menu "Method" appears immediately, see below), or
- the titrator interrupts the **Titration** function right before the end, when the condition specified for the parameter **Stop for reevaluation** is fulfilled (see **Evaluation criteria**, Section 2.3.12.2). In this case the following mask is displayed:

Reevaluation necessary

Titration [1]

neq = 0

(specified condition)

- Confirm this message with **RUN**. The following selection menu appears:

DL77/DL70ES	
	see Section
<u>Method</u>	
Add	3.1/3.13.1
Continue	3.4
Sample data	3.7
Display	3.9
Stirrer	3.10
Records	3.11
Calculations	3.12
Method data	3.8
Modify method ^{a.}	3.6
Reevaluation ^{a.}	3.5
Record data	3.8.1

DL67	
	see Section
<u>Method</u>	
Continue	3.4
Sample data	3.7
Display	3.9
Stirrer	3.10
Records	3.11
Calculations	3.12
Method data	3.8
Modify method ^{a.}	3.6
Reevaluation ^{a.}	3.5
Record data	3.8.1

^{a.} If you interrupt a METTLER method, the menus "Modify method" and "Reevaluation" are missing. This also applies to methods stored only in the computer (see *Method ID*, Section 3.1).

- Select **Reevaluation**. The following parameter mask appears:

Reevaluation
Threshold
EQP range
Steepest jump only
Buffer potential 1
Buffer potential 2

- Modify the appropriate parameters and quit the mask with **EXIT**.

The following selection menu appears:

Save
Permanent
Temporary
No

Permanent: The modified values of this **Titration** function are saved permanently in this method.

Temporary: The modified values of this **Titration** function are saved only until the end of the current method.

No: You save none of the changes.

If you confirm "Permanent" or "Temporary" with **RUN**, the titrator will reevaluate the titration curve. The printer simultaneously prints out this **Titration** function with the modified parameters. The selection menu "Method" reappears. (If your modifications still satisfy the specified condition, the message "Reevaluation necessary" reappears.)

- Confirm **Continue** with **RUN**.

On the display appears

- the representation of the **Titration** function shown before the interruption (if additional dispensing, for example, is necessary, due to the changed parameters) or
- the next function the titrator must process.

Note: To evaluate the consequences of your modification, you can

- inspect the curve or table of measured values and print it out (*Display or Records* menu, see Sections 3.9 and 3.1 1).
- check the new raw results of the **Titration** function by assigning these to Rx or Cx, e.g. Rx = VEQ oder Cx = VP1 (*Calculations* menu, see Section 3.12).

3.6 Modify current method

You have interrupted the current method and selected "Modify method". You are shown all method functions. In these functions you can modify only the **numeric** parameter values, no other parameter values or names can be selected or modified.

- Quit the parameter mask, in which you have modified a value with **EXIT**.
- To continue the titration, press **<index + A>**.

The following selection menu appears:

Save
Permanent
Temporary
No

Permanent: The modified values are saved permanently in this method.

Temporary: The modified values are saved until the start of the next titration method.

No: You save none of the changes.

After confirmation of a parameter, the selection menu "Method" reappears.

- Confirm **Continue** with **RUN**.

The representation shown before the interruption reappears on the display and at the same time, the printer automatically prints out the entire method with the modified values.

Note: In the sample determination that is interrupted, the changes apply only to the functions that the titrator has not yet begun to process. Exceptions are the **Titration** and **pH/mV-stat** functions.

1st example: **Dispense** function

If you interrupt the method during this function and change the volume then continue the method, the titrator dispenses the difference of the volume defined in the original method.

Example: defined volume →	5 mL
dispensed at interruption →	2 mL
modified volume →	7 mL
"Continue method": the titrator dispenses →	3 mL

2nd example: Titration function

If you interrupt the method during this function, with an EQP titration you can modify, for example, the value for the maximum volume or the potential value for the titration termination if this termination criterion has been specified within the method.

With an EP titration you can modify, for instance, the time for the delay if the titrant addition *Continuous* has been specified in the method.

3rd example: pH/mV-stat function

If you interrupt the method during this function, you can modify, for example, the value for the control range, the volume for the minimum consumption or the time for the specified termination criteria.

You can not change the numeric parameter values of the **Sample** function!

3.7 Sample data

You can enter the weight or volume, the ID2, and the correction factor for all samples of the methods stored in the method list before and during a current method (see also Section 3.1.2: *Notes on method and sample data mask*).

Before starting a method:

- Select the method in the method list (**not** valid for the **DL67**).
- Select **Sample data** from the selection menu: The sample data list appears.

During a current method:

- Press **<index + S>**: The sample data list of the current method appears (see example of a pH-stating on the following page).

No.	ID2	Wt./vol.
1/1		0.0000 g
1/2		0.0000 g
1/3		0.0000 g

You may enter the identification ID2 and the correction factor f, whereas you must enter the weight or volume for each individual sample (see Section 3.1.1).

- Select the first line of this mask (sample 1 of the first **Sample** function): The sample data mask appears in which you enter these parameters:

<u>Sample data</u>	
Sample No.	1/1
ID2	G/324
Weight m [g]	0.1456
Minimum [g]	0.1
Maximum [g]	0.2
Correction factor f	1.0

If you confirm a parameter with **RUN**, the mask for the second sample (No. 1/2) appears, then that of the third sample.

If no method is running, either

- press **RUN**: The selection menu "Method" appears, or
- press **<index + A>**: The method list appears.

For a running method,

- press **<index + T>**: The function the titrator is currently processing appears.

The procedure for entering sample data from other methods during a titration is described in Section 3.13.3.3.

The following example shows the sequence of a pH-stating in which the titrator should start the stating **immediately** after sample addition, meaning that the sample data are entered later.

↓

Sample data	
Sample No.	1/1
ID2	
Weight m [g]	0.0
Minimum [g]	0.0
Maximum [g]	0.2
Correction factor f	1.0

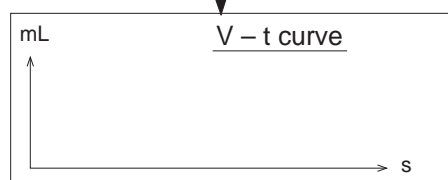
The titrator processes the **Sample** function.

- Confirm the sample data mask with **RUN**, without entering the weight.

↓

Current sample	
No. 1/1	
Method/ ID1	KM58/Ger.
ID2	

- Add the sample and confirm immediately with **RUN**.



The titrator starts the pH-stating; you have specified the V - t curve for its representation in the method.

↓

[] + [S]

- Press this key combination.

↓

No.	ID2	Wt./vol.
1/1		0.0000 g
1/2		0.0000 g
1/3		0.0000 g

The sample data list appears.

- Confirm this line with **RUN**.

↓

Sample data	
Sample No.	1/1
ID2	G/324
Weight m [g]	0.1456
Minimum [g]	0.0
Maximum [g]	0.2
Correction factor f	1.0

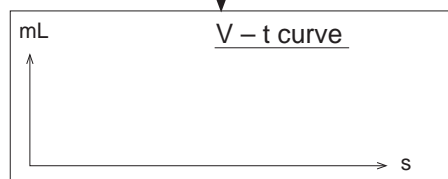
The sample data mask appears.

- Enter ID2 and weight.

↓

[] + [T]

- Press this key combination.



If the stating is still running, the V - t curve of the **pH/ mV-stat** function is displayed again. Otherwise the function the titrator is processing appears.

- Notes:
- a. If, for instance, the titration beaker containing sample No. 1 has fallen over, reselect the first line of the sample data list and overwrite weight or volume with the new value in the sample data mask.
 - b. During the titration you can modify only the data of the samples that have not yet been titrated.
 - c. You can **not modify** sample data of a method already executed.
 - d. With <**index + S**>, the sample data list for the **current** method is always shown. If no method is running, the sample data list of the method list's **first** method is shown.

3.8 Method data

You can change the method data **before** starting the method.

You can change the number of samples **during** a current method **before** the titrator processes the **Statistics** function.

- If you increase the number, the corresponding lines are added at the end of the sample data list.
- If you decrease the number, the corresponding lines are deleted at the end of the sample data list.

You can then enter the data of the added number of samples in the **Sample data** menu (see Section 3.7).

Before starting a method:

- Select the method in the method list, see Section 3.13 (**not** valid for the **DL67**).
- Select **Method data** from the selection menu: The method data list appears.

During a current method:

- Press **EXIT** and select **Method data**: The method data list of the current method appears, e.g.:

No	ID1	Samp.	Stand
1	Ca ²⁺	3	Stand 1

If one method has two or more **Sample** functions, these will also be listed, such as:

No	ID1	Samp.	Stand
1	Ca ²⁺	3	Stand 1
2	Mg/Ca	5	Stand 1

If you confirm the first line (No. 1) with **RUN**, the method data mask appears:

Method data	
Sample function No.	1
ID1	Ca ²⁺
Number samples	3
Titration stand	Stand 1
Temperature pC]	25.0
Temperature sensor	Manual
Molar mass M	110.99
Equivalent number z	2

- Modify the appropriate parameters.

When no method is running, either

- press **RUN**: The selection menu "Method" appears, or
- press <**index + A**>: The method list appears.

During a current method,

- press <**index + T**>: The function the titrator is currently processing appears.

Note: In order to modify the method data from other methods while running a method,

- press <**index + A**>: The method list appears.
- Confirm the desired method with **RUN** and select **Method data** in the selection menu.

3.8.1 Record data

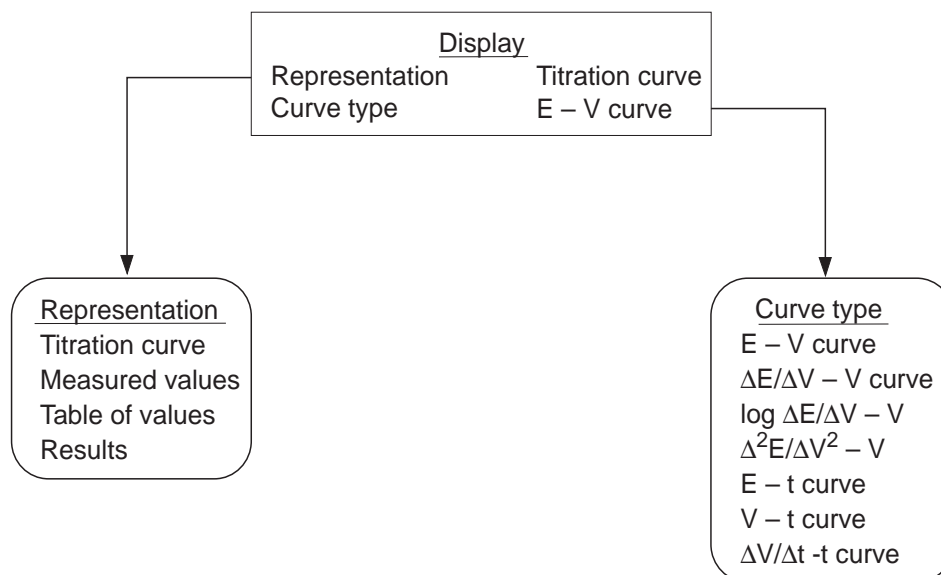
You can check all entered sample data together with the most important associated method data by requesting a record.

- Confirm **Record data** with **RUN**: All sample data stored in the memory are printed out.

3.9 Display

This menu is used for the following tasks:

- to select the parameters *titration curve*, *table of measured values* or *measured values* that the titrator can represent on the display if it executes functions or auxiliary functions that generate the necessary experimental data.
- to again follow the titration progress on the display if you have previously faded it out.
- to view the titration curve or table of measured values of the last sample once again at the end of its determination.
- to view the results that the titrator has generated during a titration method up to this time or to fade in the list of all results again at the end of the method.



Note: The key combination <index + T> returns the titration sequence to the display **during** a titration if you have faded it out beforehand.

If you select **Display**, you are shown the parameter mask (default representation)

<u>Display</u>	
Representation	Titration curve
Curve type	E - V curve

3.9.1 Representation

With this menu you can specify whether the measured values, the table of measured values or a titration curve should appear during the titration. **Titration curve** is stored as the default parameter.

Since the display has room for only one type of representation, you can select the one you wish to follow or view **before**, **during** or **after** the titration (see selection menu "Method" in Sections 3.1.3, 3.3 and 3.4).

You can change these three representations on the display **during** and **after** a titration with the aid of the **Cursor** keys (\leftarrow / \rightarrow).

Note: If you have attached a terminal (see *Peripherals*, Section 1.8.3), measured values, table of measured values and the titration curve appear on the screen **simultaneously**.

- Select from the selection menu between
Titration curve
Measured values
Table of values
Results
- 1. Titration curve: The curve selected in the **Curve type** menu is displayed (see next section).
- 2. Measured values: The measured value pair V[mL] and E (the specified unit of measurement) is displayed.
- 3. Table of measured values: The measured value pair V [mL] and E (the specified unit of measurement) is displayed with the last five pairs of values.
- 4. The list of the results generated up to this point appears on the display (only if you did interrupt the method!).
- To follow the selected representation, press **RUN**.

3.9.2 Curve type

With this menu you can specify the curve that can be generated by the **Titration** or **pH/mV-stat** function. The **E - V** curve is stored as the default curve.

- Select from the selection menu between:

E - V curve	Potential vs volume
$\Delta E / \Delta V$ - V curve	1st derivative (potential vs volume)
$\log \Delta E / \Delta V$ - V (curve)	1st derivative (potential vs volume, logarithmic)
$\Delta^2 E / \Delta V^2$ - V (curve)	2nd derivative (potential vs volume)
E - t curve	Potential vs time
V - t curve	Volume vs time
$\Delta V / \Delta t$ - t curve	1st derivative (volume vs time)

- To follow the selected curve type on the display, press **RUN**.

Notes: a. If you wish to change the curve type during a determination,

- fade out the titration with **EXIT**,
- select the *Display* menu.
- select the desired curve type and confirm with **RUN**: The selected curve appears.

b. If you wish to view, for instance, the table of measured values or the titration curve once again at the end of a sample determination,

- fade out the titration with **EXIT**,
- in the selection menu, confirm **Interrupt** with **RUN**,
- press **<index + T>** and select with the aid of the **Cursor keys** (\leftarrow / \rightarrow) between
 - the last measured value pair.
 - the last five tabulated values; with the aid of the arrow key you can follow the table of measured values back to the initial values, however.
 - the selected titration curve.

c. If you wish to view the table of measured values or the titration curve once again at the end of a method,

- press **<index + T>** (see Note b.)

3.10 Stirrer

You can change the stirrer speed during a currently running or interrupted method (see Sections 3.3 and 3.4).

If you confirm **Stirrer** with **RUN**, the following parameter mask appears (example):

<u>Stirrer</u>	
Speed [%]	80
Status	On

If you change the speed and confirm it with **RUN**, the stirrer then stirs at the specified speed until the next **Sample** or **Stir** function.

If you wish to switch off the stirrer, select **Status** with **SEL** → **Off**. It remains switched off

- until the next **Sample** or **Stir** function or
- until you select status **On**.

3.11 Records

With the aid of this function you can generate an additional record on the printer at the end of every sample determination. For this, you must interrupt the method (see Section 3.4)

If you select this menu, the following parameter mask appears:

- | | |
|---------------------------------------|--|
| 1. Short form method | Would you like a record of the method in short form?
Select "Yes" with SEL . |
| 2. Sample data | Would you like a record of all sample data (ID1, ID2, molar mass, equivalent number, weight/volume, correction factor) of the last sample?
Select "Yes" with SEL . |
| 3. Raw results last sample | Would you like a record of all raw results such as VEQ or VDISP of the last sample?
Select "Yes" with SEL . |
| 4. Results last sample | Would you like a record of all results of the last sample?
Select "Yes" with SEL . |
| 5. All results | Would you like a record of all previously generated results?
Select "Yes" with SEL . |
| 6. Table of measured values | Would you like a record of the table of measured values of the last sample?
Select "Yes" with SEL . |
| 7. E - V curve | Would you like a record of the titration curve potential vs volume of the last sample?
Select "Yes" with SEL . |
| 8. $\Delta E/\Delta V$ - V curve | Would you like a record of the 1st derivative of the titration curve potential vs volume of the last sample?
- (The ordinate scale is linear.)
Select "Yes" with SEL . |
| 9. $\log \Delta E/\Delta V$ - V curve | Would you like a record of the 1st derivative of the titration curve potential vs volume of the last sample?
- (The ordinate scale is logarithmic.)
Select "Yes" with SEL . |

-
- | | |
|---|---|
| 10. $\Delta^2 E / \Delta V^2$ - V curve | Would you like a record of the 2nd derivative of the titration curve potential vs volume of the last sample?
- (The ordinate scale is linear.)
Select "Yes" with SEL . |
| 11. E - t curve | Would you like a record of the titration curve potential vs time of the last sample?
Select "Yes" with SEL . |
| 12. V - t curve | Would you like a record of the titration curve volume vs time of the last sample?
Select "Yes" with SEL . |
| 13. $\Delta V / \Delta t$ - t curve | Would you like a record of the titration curve of the 1st derivative volume vs time of the last sample?
Select "Yes" with SEL . |

If you confirm one of the parameters with **RUN**, the data are printed out.

3.10 Calculations

With the aid of this function you can perform additional calculations at the end of every sample determination. For this, you must interrupt the method (see Section 3.4). The result appears on the display; it can not be printed out.

If you select this menu, the following parameter mask appears:

```
Calculations
Result name
Formula   Rx =
Constant  Cx =
Result unit
Decimal places
```

1. Enter the result name.
2. Select the formula from the recommendation menu or enter the one you need for your calculation. (**Caution:** Always enter Cx as constant!)
3. Select the constant from the recommendation menu or enter the one you need for your calculation.

Note: If you press **HELP** you are shown for

Formula the most common formulae listed with the possible units and for

Constant the most common constants with the corresponding units.

4. Select the result unit from the recommendation menu or enter the one you need for your calculation.
5. Enter the number of decimal places that you desire in the result.

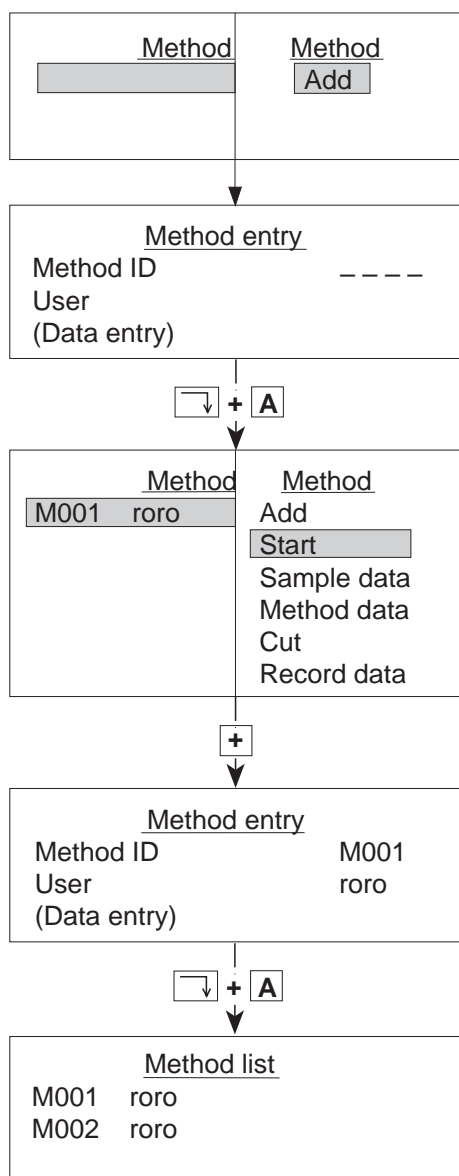
If you confirm one of the parameters with **RUN**, the result will be displayed.

3.13 Method list

You can add a maximum of 10 methods, each with at most 60 samples, to the method list. These will be stored with the modified method data and entered sample data. They remain stored when the titrator is turned off; the data from an executed method, however, will be deleted. You can change the order of the methods.

DL67: The titrator can load only one method (see Section 3.1).

3.13.1 Filling the method list



If the method list is empty, it appears together with the selection menu.

– Confirm **Add** with **RUN**.

– Enter or modify the method identification, your name, and the data entry, if necessary.

– Press this key combination.

The method list with the entries and the selection menu appears.

– Press this key.

The names you entered for the first method are in the method entry mask. You can simply change the method ID.

– Press this key combination.

The method list appears again, etc.

- Notes:
- If no method with the specified identification is stored in the titrator and you have installed a computer, the method will be requested from the computer and added to the method list (see Operating Instructions provided with the RS option).
 - You can modify or enter method and sample data
 - immediately (see Section 3.1)
 - before starting the method (see Section 3.1)
 - during a current method (see Sections 3.7 and 3.8).

3.13.2 Modifying the method list

You can cut (delete) an added method and paste it in at another location.

3.13.2.1 Cut

Example: Method list
MOO1 roro
M002 roro
CALC roro
KM58 roro

- Select the method (e.g KM58) and press the <=> key (minus sign).
The 4th method together with its sample data is cut (deleted), however, it is stored in a buffer memory so that you can paste it elsewhere.

3.13.2.1 Paste

You would now like to position the cut method KM58 with its sample data on the top of the method list:

- Select method "M001" and press the <=>key (equals sign): The former 4th method is now the 1st method.

Method list
KM58 roro
MOO1 roro
M002 roro
CALC roro

Caution: Bear in mind the sequence of the sample beakers if you execute these commands!

3.13.3 Processing the method list

If you confirm one of the methods with **RUN**, you can start, e.g. activate, it with the selection menu below:

Start

Single method

List once

List continuous

3.13.3.1 Single method

Select **Single method** if you

- have stored only one method or
- wish to perform only that method selected among several stored methods.

The executed method is marked with an asterisk (*). As soon as you start another method, all data from the executed method will be deleted. If you select the executed method and confirm **Start**, the titrator will reload this method (see *Restarting a previously executed method*, Section 3.1.4).

3.13.3.2 List once

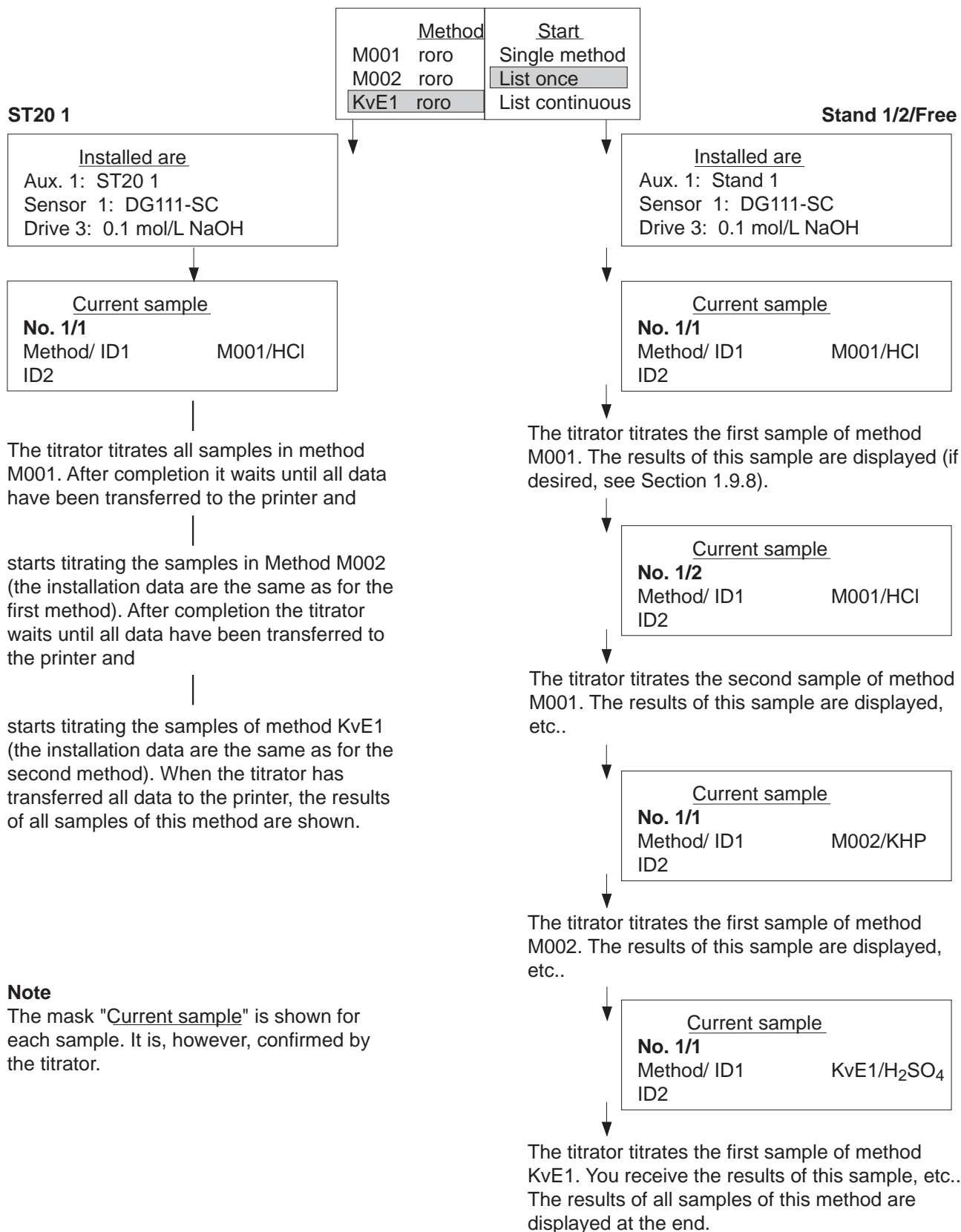
Select **List once** if you wish to perform several stored methods in succession. Here it is immaterial which method you have selected: The titrator begins with the method at the start of the method list.

- The method currently running is marked in the method list with ">".
- At the end of a processed list, only the final method, marked with an asterisk (*), will be saved. It will be deleted once you start a newly entered list.

Notes: a. If you are working at titration stand 1, 2 or a free stand, the sample data mask appears only if you have not yet entered the weight (volume) of the samples. You must confirm the mask "Current sample" for every sample.

- b. If you use the auto stand, the mask "Current sample" will not appear. This means that the next sample determination will be started **without** a **RUN** confirmation. Thus you can assign the sample changes for each method to a robot.
- c. If you have attached the sample changer, the titrator titrates the samples of all methods without the need for you to intervene. A requirement here is that
- all sample data have been entered,
 - the methods use the same resources or that their installation data are specified on different drives (titrants) and inputs (sensors), see comparison of the titration sequences on the next page.

Comparison of the titration sequence for several methods at the titration stand ST20 1 (ST20 2) or at Stand 1, 2 or a free stand.



If the titrator should process methods, that use the same burette drives or sensor inputs, on the ST20A (ST20) sample changer in succession, you have to intervene:

Method	Start
M001 roro	Single method
M002 roro	List once
CALC roro	List continuous



Installed are
Aux. 1: ST20 1
Sensor 1: DG111-SC
Sensor 2: DG141-SC
Drive 3: 0.1 mol/L NaOH
Drive 3: 0.1 mol/L AgNO ₃



Current sample
No. 1/1
Method/ ID1 M001/HCl
ID2



The titrator titrates all samples of method M001. After completion it waits until all data have been transferred to the printer and



starts titrating the samples of method M002 (the installation data are the same as for the first method). When the titrator has transferred all data to the printer, the following request appears:

Change resources
Drive 3: 0.1 mol/L AgNO ₃



The titrator starts to titrate the samples of method CALC. When all data have been transferred to the printer, the results of all samples of this method will be displayed.

The installation data of **all** methods are displayed.

This mask appears for all samples but is confirmed by the titrator.

- Change the titrant.
 - Attach the DM141-SC sensor and insert it.
- This is not requested, as the sensor is installed at sensor input **2**.

3.13.3.3 List continuous

Select **List continuous** if you wish to repeat the listed methods once or several times.

- When the titrator has completed the first method, it moves it to the end of the method list. All data of the method data mask remain stored (ID1, number samples, titration stand, temperature, temperature sensor, molar mass, and equivalent number). You can modify these (see Section 3.8).

All data of the sample data mask are deleted (ID2, weight/volume and correction factor) as soon as the titrator starts the second method.

If you have defined a fixed volume in the **Sample** function, this remains stored.

Enter all data that you have to modify and/or new data you have to save during the titration of the following methods (see scheme on the next page).

- When the titrator has completed the second method, it also moves it to the end of the method list, etc..
- The method currently running is marked in the method list with ">".

You close the **List continuous** procedure with **RESET**.

If the titrator has no sample data for the method to be processed, the sample data mask will be shown.

- Press **RESET**. In the menu that appears (example)

Terminate

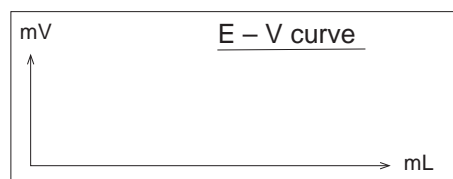
ANALYSIS A* ; Method: RM35

(* appears only with the DL77)

- confirm "Method: RM35" with **RUN** and quit the mask with **EXIT**. **List continuous** is terminated definitively.

This most recently "started" method is marked with an asterisk (*) and remains stored, together with all "shifted" methods, without sample data. Should you now restart **List continuous**, the titrator would begin with method RM35.

Example showing how to enter sample data for methods while **List continuous** is being processed.



↓ + A

Method list	
> M001	roro
HSO4	roro
HPO4	roro

Method	Method
> M001	roro
HSO4	roro
HPO4	roro
	Add
	Sample data
	Method data
	Cut
	Record data

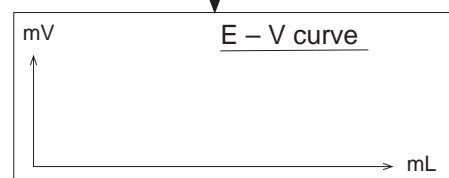
No.	ID2	Wt./vol.
1/1		0.0000 g
1/2		0.0000 g
1/3		0.0000 g

Sample data	
Sample No.	1/1
ID2	PS/389A
Weight m [g]	2.4871
Minimum [g]	2.0
Maximum [g]	3.0
Correction factor f	1.0

Sample data	
Sample No.	1/2
ID2	PS/389B
Weight m [g]	2.543
Minimum [g]	2.0
Maximum [g]	3.0
Correction factor f	1.0

Sample data	
Sample No.	1/3
ID2	PS/389C
Weight m [g]	2.2671
Minimum [g]	2.0
Maximum [g]	3.0
Correction factor f	1.0

↓ + T



Method M001 is running. To enter the sample data for method HPO4,

– press this key combination.

– Confirm the last method with **RUN**.

– Confirm **Sample data** with **RUN**.

– Confirm first line with **RUN**.

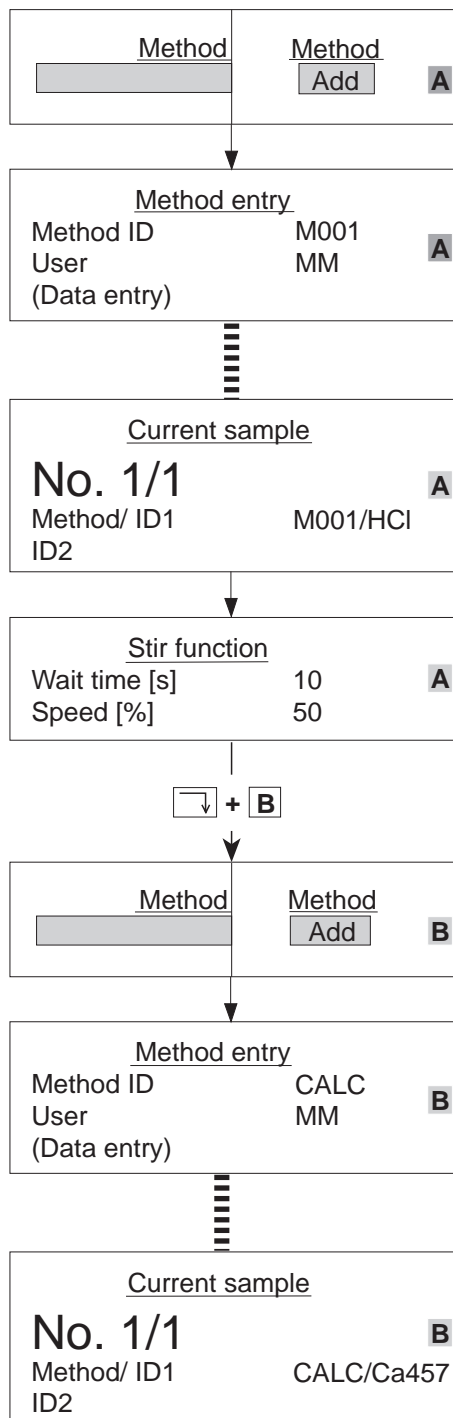
- Enter the second identification, the correction factor (if necessary) and the weight and confirm with **RUN**: the sample data mask for the second, then for the third sample appears.
- To follow the titration course of the method again, press <Index + T>.

3.14 Parallel titrations with the DL77

Two Analysis menus **A** and **B** exist for the simultaneous execution of methods. They are equivalent in terms of their build-up and functionality, meaning that you can start titration under ANALYSIS A or ANALYSIS B.

You can store 10 methods with a maximum of 60 samples in **both** method lists.

Procedure (example):



ANALYSIS A is selected.

– Confirm **Add** with **RUN**.

– Enter the method identification and your name, etc..

If you use the titration stand 1 or 2,

– confirm this mask with **RUN**.

If you use titration stand ST20 1 or 2, the titrator confirms this mask.

The method M001 is processed. You can switch over to ANALYSIS B.

– Press this key combination.

ANALYSIS B is selected.

– Confirm **Add** with **RUN**.

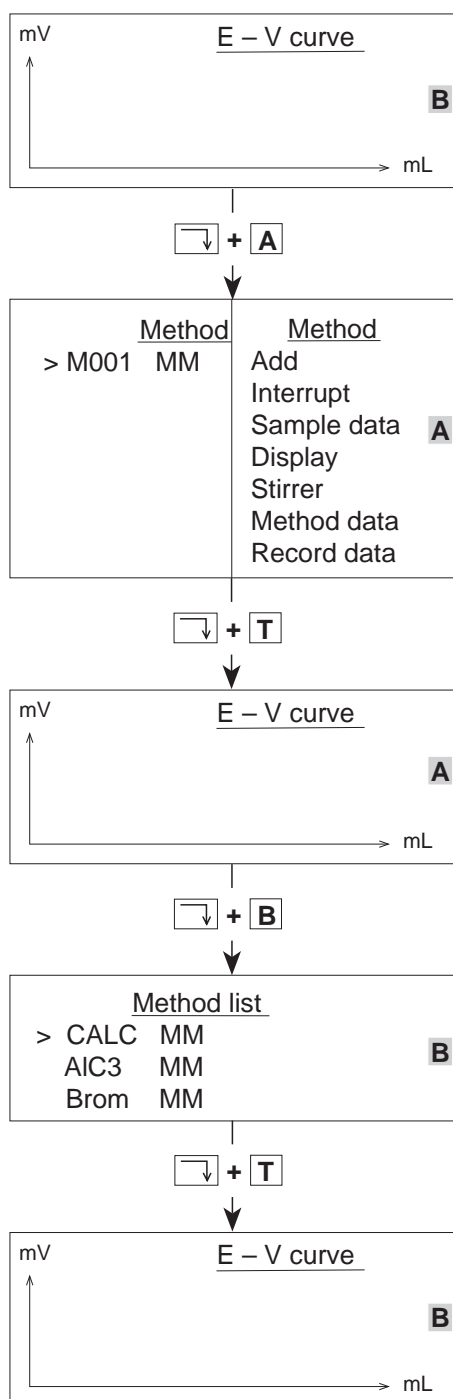
– Enter the method identification and your name, etc..

If you confirm this mask with **RUN**, the method CALC is processed.

3.14.1 Notes on parallel titrations

- a. All resources (titrants, sensors, auxiliary instruments, etc.), i.e., all the associated drives, inputs and outputs that are reserved for **active** methods under ANALYSIS A can not be used for methods under ANALYSIS B! **Active**
 - is either a running **Single method** or
 - are all methods that have been started with the commands **List once** or **List continuous!**
- b. Parallel titrations can be, for example, performed with titration stand ST20 1 (Sample series) and Stand 1 (Karl Fischer).
Using two connected sample changers you can, for example, determine two sample series in parallel (ST20 1 and ST20 2, see Section 1.8.4).
- c. The running method's functions **Record** are executed sequentially:
 - Should the titrator first process a **Record** function under ANALYSIS B, then these data will be printed out first.
 - If the titrator reaches a **Record** function under ANALYSIS A a short time thereafter, it will wait with the method processing until the data under ANALYSIS B have been printed out.
- d. You can change from one Analysis menu to the other at anytime using the key combination <index + A> or <index + B> respectively (see example on the next page).
- e. The **Sync** function can be used to coordinate methods in the method lists of Analysis menus A and B by their chronological sequence. For this you need the RS option as well as an RS short circuit plug (see *Sync*, Section 2.3.20 and Operating Instructions provided with the RS option).

3.14.2 Changing the Analysis menus



In ANALYSIS B the Titration function of the CALC method is processed.

- To switch to ANALYSIS A, press this key combination.

The method list with the selection menu ("method faded out") appears.

You can select or execute all menus or commands.

- In order to follow the running method M001 on the display, press this key combination.

- To switch back to ANALYSIS B, press this key combination.

The method list appears if more than one method is stored. You can select one of the methods in order to modify method or sample data using the selection menu.

- To follow the still running method CALC on the display immediately, press **<index> + T**.

3.15 Sample series with 2 sample changers

If you have connected two sample changers, you can sequentially titrate maximally 40 samples from one or several methods "unattended". The titrator is told to change the titration stand - from ST20 1 to ST20 2 or vice versa - only via the **Sample** function of a method (see Section 2.3.2).

Example for **one** method with 40 samples: Only the functions in which parameters **must** be modified are shown. Parameters in the **Stir** and **Rinse** functions (these are not shown in the scheme) should not be modified!

SAMPLE	Number samples	20
	Titration stand	ST20 1
	etc.	

The first 20 samples are titrated using titration stand ST20 1.

TITRATION	Titrant	HCl
	Concentration [mol/L]	0.1
	Sensor	DG111-SC
	etc.	

HCl and **DG11 1-SC** are stored as standard resources.

CALCULATION	Results	Base
	Formula	$R=Q \cdot C/U$
	Constant	$C=M/z$
	etc.	etc.

The content in g/L is calculated.

STATISTICS	Ri (i=index)	R1
	etc.	

After 20 sample determinations, their mean value is calculated.

SAMPLE	Number samples	20
	Titration stand	ST20 2
	etc.	

The titrator is told by the 2nd *Sample* function to titrate the next 20 samples using titration stand ST20 2.

TITRATION	Titrant	HCl-2
	Concentration [mol/L]	0.1
	Sensor	DG111-2
	etc.	

HCl-2 must be added to the list of titrants and **DG111-2** to the list of sensors (see Section 1.1.3 and 1.2.3).

CALCULATION	Results	Base
	Formula	$R2=Q[2] \cdot C/U$
	Constant	$C=M/z$
	etc.	etc.

The result of the 2nd *Calculation* function must receive **2** as the index for **Q**: mmol consumption in the **2nd Titration** function (see Section 8.2).

STATISTICS	Ri (i=index)	R2
	etc.	

The mean value of the **2nd Titration** function is calculated.

CALCULATION	Results	Base
	Formula	$R3=(\bar{x}+\bar{x}[2])/2$
	Constant	
	etc.	

The mean value of all titrated samples is calculated with the 3rd *Calculation* function.

If you wish to run several methods on both sample changers with **List once**, one of the methods must be used to initiate the change from one stand to the other in each case. An example:

Method list	
HAC1	dabru
HSO4	dabru
CALC	dabru

The first two methods are acid content determinations, thus neither titrant nor sensor must be changed.

Method data	
Sample function No.	1
ID1	AA23
Number samples	10
Titration stand	ST20 1
Temperature [°C]	25.0
Temperature sensor	Manual
Molar mass M	60.05
Equivalent number z	1

Ten samples from method HAC1 are titrated using titration stand ST20 1.



Method data	
Sample function No.	1
ID1	SA/666
Number samples	7
Titration stand	ST20 1
Temperature [°C]	25.0
Temperature sensor	Manual
Molar mass M	98.07
Equivalent number z	2

Seven samples from method HSO4 are titrated.



Method data	
Sample function No.	1
ID1	Ca/k34
Number samples	15
Titration stand	ST20 2
Temperature [°C]	25.0
Temperature sensor	Manual
Molar mass M	110.99
Equivalent number z	2

Titration stand ST20 2 is activated by the **Sample** function of method CALC. The 15 samples are titrated.

Contents	Page
4. AUXILIARY FUNCTIONS	4-3
4.1 Burette	4-6
4.1.1 Rinse burette	4-6
4.1.2 Rinse tip	4-7
4.1.3 Dispense	4-7
4.1.4 Dispense continuously	4-8
4.1.5 Manual titration	4-9
4.2 Stirrer	4-11
4.3 Sensor	4-12
4.4 Temperature	4-14
4.5 Sample changer	4-16
4.5.1 Lift	4-16
4.5.2 Turntable forward	4-17
4.5.3 Turntable backward	4-17
4.5.4 Rinsing pump	4-18
4.5.5 Rinsing pump manual	4-18
4.5.6 Dosing pump	4-19
4.5.7 Dosing pump manual	4-20
4.5.8 Dispenser	4-20
4.6 Auxiliary instrument	4-21
4.6.1 Time-controlled	4-21
4.6.2 Manual	4-22
4.7 Calibration of the temperature sensors	4-23
4.8 Offset adjustment of the sensor inputs	4-24

4. AUXILIARY FUNCTIONS

With the aid of this menu you can rinse burettes, perform manual titrations, start the stirrer, measure the pH or temperature of solutions, operate the sample changer manually and pump solvents. These auxiliary functions are independent of a titration method, but they can support or expand it.

The menu:

	<u>Auxiliary functions</u>
1	Burette
2	Stirrer
3	Sensor
4	Temperature
5	Sample changer
6	Auxiliary instrument
7	Calibration temperature sensors
8	Offset adjustment sensor inputs

As with all menus of the titrator you can select this menu and its submenus by moving the selector bar and confirming with **RUN**.

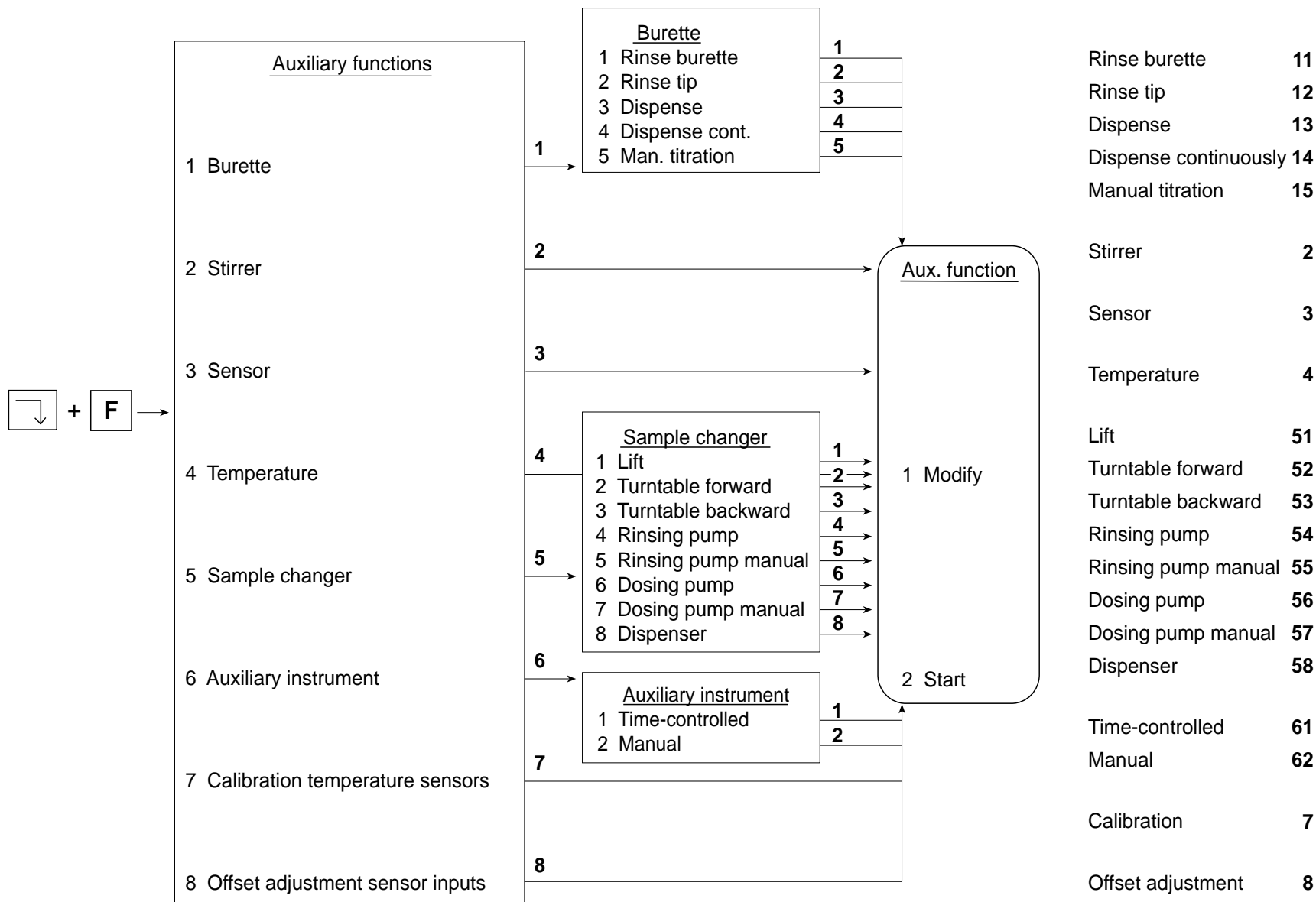
Since you have to select the menu and the auxiliary functions frequently, there is a faster way:

- Use the key combination <index + F> to obtain the menu.
- For the auxiliary functions themselves, numeric keys take over the function of the selector bar and the **RUN** key. You press only the key with the function number that precedes every auxiliary function.

If you know the function number you can already press the corresponding keys in any menu (see menu representation on the following page).

Note: The lines "4 Temperature" and "7 Calibration temperature sensors" appear only if you have inserted a temperature option; the line "5 Sample changer" appears when you have installed an ST20A (ST20), see Sections 11.1.7 and 1.8.4.

Menu tree with function numbers



Notes: a. With the following key combinations, the two auxiliary functions **Rinse burette** and **Rinse tip** can be executed even faster;

<**Index+U**> → Rinse burette

Press **1**, **2**, **3**, or **4** to select the burette drive: The burette is rinsed.

<**Index+Y**> → Rinse tip

Press **1**, **2**, **3**, or **4** to select the burette drive: The burette tip is rinsed.

b. Once one of the auxiliary functions has been executed, you can quit the selection menu

Aux. function

1 Modify

2 Start

- with **EXIT**: The next higher menu will appear, or
- with a key combination (index + letter): The selected menu will appear.

c. Should several auxiliary functions be active, such as "Sensor" at titration stand 1 and "Stirrer" and "Burette/Dispense" at titration stand 2, and you would like to stop all three, press **RESET**. The following display will appear:

Terminate

Sensor Sensor 1

Stirrer Aux. 1

Dispense Drive 3

All functions are now **interrupted**. To terminate them

- select "Sensor ..." and confirm with **RUN**,
- confirm "Stirrer ..." with **RUN**,
- confirm "Dispense ..." with **RUN** and
- quit the mask with **EXIT**.

You are shown the display in which you have pressed **RESET**.

4.1 Burette

If you rinse burettes or burette tips or wish to dispense a certain volume, select this auxiliary function. You can press **1** in the *Auxiliary functions* menu, when in any other menu **<index + F>**, then **1**.

You are shown the following submenu:

1	Rinse burette	Drive 3
2	Rinse tip	Drive 3
3	Dispense	Drive 3
4	Dispense cont. (inuously)	Drive 3
5	Man. (ual) titration	NaOH DG111-SC

Caution: Only two burette drives can be **active simultaneously!**

Burettes reserved for a certain method cannot be selected. This applies to

- the burettes in use for a current method and
- all burettes that will be used in the methods that have been activated with the commands **List once** or **List continuous** (see Sections 3.13.3.2 and 3.13.3.3).

4.1.1 Rinse burette

To execute this function, from within any menu

- press the key combination **<index + U>** and
- enter the burette drive: **1, 2, 3, or 4**:

The titrator delivers 1 burette volume. The piston of the burette moves below the zero position so that air bubbles that "hide" in the stopcock are sucked out and then expelled.

- To stop the operation, press **RUN** or **3**.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.1.2 Rinse tip

To execute this function from within any menu

- press the key combination **<index + U>** and
- enter the burette drive: **1**, **2**, **3**, or **4**:

5% of the burette volume is dispensed to avoid possible diffusion effects with the solvent or crystallization in or at the tip of the dispensing tube.

You can repeat the operation with **RUN**.

- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.1.3 Dispense

With the aid of this function you can dispense a fixed volume. To execute it, press function number **3**. If you press **1 3** in the *Auxiliary functions* menu you are immediately shown the selection menu.

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameters whose value or name you can change. If you press, **<index + F> 1 3 1** when, e.g. in the main menu, you are shown the parameter mask immediately.

1. Select the burette drive from the selection menu.
2. Select the burette volume from the selection menu.
3. Enter the volume [mL].

If you confirm one of the parameters with **RUN**, you can start dispensing.

Start: press **2** or **RUN**.

- The titrator dispenses the specified volume.

You can stop dispensing with **RUN** or **3**.

- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.1.4 Dispense continuously

With the aid of this function you can dispense continuously and follow the change in potential with the dispensing on the display. To execute the function, press function number **4**. If you press **1 4** in the *Auxiliary functions* menu you are immediately shown the selection menu:

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameters whose value or name you can change. If you press **<index + F> 1 4 1** when, e.g. in the main menu, you are shown the parameter mask immediately.

1. Select the burette drive from the selection menu.
2. Select the burette volume from the selection menu.
3. Enter the rate [mL/min] at which dispensing should be performed.
4. Select the sensor from the recommendation menu or enter the one you have installed.
The sensor acquires the measured values in the unit of measurement you have installed (see Section 1.2.2).
5. Select the titration stand from the selection menu.
6. Enter the speed of the stirrer [0 – 100%]:
 - 0 → stirrer is inactive;
 - 100 → stirrer operates at maximum speed.
7. Enter the temperature [°C] of the solution to be measured (if you have not attached a temperature sensor).
8. Select the temperature sensor from the selection menu.

If you confirm one of the parameters with **RUN**, you can start dispensing.

Start: press **2** or **RUN**.

- The titrator dispenses continuously and acquires the potential values of the solution undergoing change. You can follow the measured values mL and mV (pH,...) on the display (in the selection menu "Aux. function" you can select the display with **4** while dispensing).
- To stop the dispensing, confirm the display with **RUN** and press **3**.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

Note: If you carry on with the continuous dispensing, but wish to process other menus in the meantime, quit the display with the appropriate key combination (index + letter).

- To follow the measured values later on the display, press **<index + F>1 4 4**.

4.1.5 Manual titration

With the aid of this function you can titrate manually and follow the change in potential with the dispensing on the display. To execute the function, press function number **5**. If you press **1 5** in the *Auxiliary functions* menu you are immediately shown the selection menu:

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameters whose value or name you can change. If you press **<index + F> 1 5 1** when, e.g. in the main menu, you are shown the parameter mask immediately.

1. Select the titrant from the recommendation menu or enter the one you have installed (see Section 1.1).
2. Enter its concentration [mol/L].
3. Select the sensor from the recommendation menu or enter the one you have installed (see Section 1.2).
4. Select the unit of measurement: "mV" or "As installed".
"As installed" refers to the unit of measurement you have defined for this sensor (see Section 1.2.2).
5. Select the titration stand from the selection menu.
6. Enter the speed of the stirrer [0 – 100%]:
 - 0 → stirrer is inactive;
 - 100 → stirrer operates at maximum speed.
7. Enter the temperature [°C] of the solution to be measured (if you have not attached a temperature sensor).
8. Select the temperature sensor from the selection menu.

If you confirm one of the parameters with **RUN**, you can start manual titration.

Start: press **2** or **RUN**.

- The titrator begins to dispense and increases the dispensing rate continuously. The left part of the display shows the measured values mL and mV, (pH,...), in the right part the following selection menu appears:

Interrupt

Terminate

Interrupt

Dispensing is interrupted by pressing **RUN**.

- The potential value will still be measured.
- **Interrupt** will be replaced in the display by **Continue**.

With **RUN** the titrator proceeds with the lowest dispensing rate, thus, addition near the end point can be controlled with the **RUN** key.

Terminate

The titration is terminated by pressing **RUN**.

- The left part of the display shows the results (volumes in mL and mmol and the potential value in the selected unit of measurement).
 - Pressing **Print** in the right part of the display enables you to print the results.
- To start with the next manual titration, press **EXIT** and confirm **Start**.
 - To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.2 Stirrer

If you wish to stir solutions to dissolve substances or to support a titration method without a *Sample* function, select this auxiliary function.

Caution: Stirrer outputs reserved for a certain method (using the titration stand specified in the *Sample* function) cannot be selected. This applies to

- the stirrer output being used in a current method and
- all stirrer outputs that will be used in the methods that have been activated with the commands **List once** or **List continuous** (see Sections 3.13.3.2 and 3.13.3.3).

If you press function number **2** (in one of the other menus **<index + F>**, then **2**), you are shown the selection menu:

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameters whose value or name you can modify. If you press **<index + F> 2 1**, when, e.g. in the main menu, you are shown the parameter mask immediately.

1. Select the titration stand from the selection menu.

The titrator now knows which stirrer it has to control (see *Titration stands*, Section 1.7).

2. Enter the speed of the stirrer [0 – 100%]:

- 0 → stirrer is inactive;
- 100 → stirrer operates at maximum speed.

If you confirm a parameter with **RUN**, you can start stirring.

Start: press **2** or **RUN**.

- The titrator begins to stir at the specified speed.

You can stop stirring with **RUN** or **3**.

- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

Note: If you wish to execute another auxiliary function (e.g. *Dispense*) or a method which utilizes **Stirrer**

- start this auxiliary function first,
- quit the selection menu with the appropriate key combination (**<index + F>** or **<index + A>**), and then
- select the other auxiliary function or start the method.

4.3 Sensor

With the aid of this function you can measure potential values of solutions (mV, pH, %T, etc.). The measured values can be printed out.

Caution: You can measure **simultaneously** with only two sensors!

Sensors reserved for a certain method cannot be selected. This applies to

- the sensors being used in a current method and
- all sensors that will be used in the methods that have been activated with the commands **List once** or **List continuous** (see Sections 3.13.3.2 and 3.13.3.3).

If you press function number **3** (in one of the other menus <**index + F**>, then **3**), you are shown the selection menu:

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameters whose value or name you can modify. If you press <**index + F**> **3 1**, when, e.g. in the main menu, you are shown the parameter mask immediately.

1. Select the sensor from the recommendation menu or enter the one you have installed (see Section 1.2).
2. Select the unit of measurement: "mV" or "As installed".
"As installed" refers to the unit of measurement you have defined for this sensor (see Section 1.2.2).
3. Select the titration stand from the selection menu.
The titrator now knows which stirrer it has to control (see *Titration stands*, Section 1.7).
4. Enter the speed of the stirrer [0 – 100%]:
 - 0 → stirrer is inactive;
 - 100 → stirrer operates at maximum speed.
5. Enter the temperature [°C] of the solution to be measured (if you have not attached a temperature sensor).
6. Select the temperature sensor from the selection menu.
7. Select record: "Yes" or "No". → Yes: the measured values are recorded.
8. Enter Δt [s] : e.g. 10. → A measured value will be recorded every 10 seconds.

If you confirm one of the parameters with **RUN**, you can measure the potential of the solution.

Start: press **2** or **RUN**.

- The titrator starts to measure the potential of the solution. The measured values appear on the display and are recorded.
- To stop the measurement, confirm the mask with **RUN** and press **3**.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

Note: If you continue the measurement but in the meantime wish to process other menus, quit the display with the appropriate key combination (index + letter). Printout of the measured values continues.

- To follow the measured values later on the display, press **<index + F> 3 4**.

4.4 Temperature

With the aid of this function you can measure temperature values of solutions (°C, °F or K). The measured values can be printed out.

Caution: Temperature sensors reserved for a certain method cannot be selected. This applies to

- the temperature sensor being used in a current method and
- all temperature sensors that will be used in the methods that have been activated with the commands **List once** or **List continuous** (see Sections 3.13.3.2 and 3.13.3.3).

If you press function number **4** (in one of the other menus <**index** + **F**>, then **4**), you are shown the selection menu:

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameters whose value or name you can modify. If you press <**index** + **F**> **4 1**, when, e.g. in the main menu, you are shown the parameter mask immediately.

1. Select the temperature sensor from the selection menu.
2. Select the unit of measurement from the selection menu.
3. Select the titration stand from the selection menu.
The titrator now knows which stirrer it has to control (see *Titration stands*, Section 1.7).
4. Enter the speed of the stirrer [0 – 100%]:
 - 0 → stirrer is inactive;
 - 100 → stirrer operates at maximum speed.
5. Select record: "Yes" or "No". → Yes: the measured values are recorded.
6. Enter Δt [s] : e.g. 10. → A measured value will be recorded every 10 seconds.

If you confirm one of the parameters with **RUN**, you can measure the temperature of the solution.

Start: press **2** or **RUN**.

- The titrator starts to measure the temperature of the solution. The measured values appear on the display and are recorded.
- To stop the measurement, confirm the mask with **RUN** and press **3**.

- To obtain the *Auxiliary functions* menu again, press <**index** + **F**>.

Note: If you continue the measurement but in the meantime wish to process other menus, quit the display with the appropriate key combination (index + letter). Printout of the measured values continues.

- To follow the measured values later on the display, press <**index** + **F**> **4 4**.

4.5 Sample changer

With the aid of this function you control the sample changer (titration stand ST201 or ST202) and its attached auxiliary units.

DL70ES/DL67: You cannot select "ST20 2", as a second sample changer can not be connected.

If you press function number **5** (in one of the other menus <**index + F**>, then **5**), you are shown the following menu:

- 1 Lift
- 2 Turntable forward
- 3 Turntable backward
- 4 Rinsing pump
- 5 Rinsing pump manual
- 6 Dosing pump
- 7 Dosing pump manual
- 8 Dispenser

4.5.1 Lift

If you wish to change the lift position, press function number **1**. If you press **5 1** in the *Auxiliary functions* menu you are immediately shown the selection menu:

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

1. Select the sample changer: "ST20 1" or "ST20 2".

If you confirm the selection with **RUN**, you can change the lift position.

Start: press **2** or **RUN**.

If you press <**index + F**> **5 1 2** when, e.g. in the main menu, you can change the lift position immediately.

- If the lift is in the topmost position, it moves to the middle position.
 - If it is in the middle position, it moves to the lowest position.
 - If it is in the lowest position, it returns to the topmost position.
- To obtain the *Auxiliary functions* menu again, press <**index + F**>.

4.5.2 Turntable forward

If you wish to move the turntable forward, press function number **2**. If you press **5 2** in the *Auxiliary functions* menu, you are immediately shown the selection menu.

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

1. Select the sample changer: "ST20 1" or "ST20 2".
2. Enter the number of positions the turntable should move forward.

If you confirm the entry with **RUN**, you can move the turntable.

Start: press **2** or **RUN**.

If you know what number is stored for the positions (and the ST20), you can, e.g. when in the main menu immediately move the turntable forward with **<index + F> 5 2 2**.

- The turntable moves forward by the specified number of positions.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.5.3 Turntable backward

If you wish to move the turntable backward, press function number **3**. If you press **5 3** in the *Auxiliary functions* menu you are shown the selection menu immediately.

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

1. Select the sample changer: "ST20 1" or "ST20 2".
2. Enter the number of positions the turntable should move backward.

If you confirm the entry with **RUN**, you can move the turntable.

Start: press **2** or **RUN**.

If you know what number is stored for the positions (and the ST20), you can, e.g. when in the main menu immediately move the turntable backward with **<index + F> 5 3 2**.

- The turntable moves backward by the specified number of positions.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.5.4 Rinsing pump

If you wish to perform rinsing for a certain time, press function number **4**. If you press **5 4** in the *Auxiliary functions* menu you are immediately shown the selection menu.

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameter mask. If you press **<index + F> 5 4 1** when, e.g. in the main menu, you are shown this mask immediately.

1. Select the sample changer: "ST20 1" or "ST20 2".
2. Enter the time [s] during which the pump attached to the sample changer should perform rinsing.
The specified time is an empirical value.

If you confirm the entry with **RUN**, you can start rinsing.

Start: press **2** or **RUN**.

- The pump rinses for the specified time.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.5.5 Rinsing pump manual

If you wish to interrupt a rinsing process manually, press function number **5**. If you press **5 5** in the *Auxiliary functions* menu you are immediately shown the selection menu.

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

1. Select the sample changer: "ST20 1" or "ST20 2".

If you confirm the entry with **RUN**, you can start rinsing.

Start: press **2** or **RUN**.

If you press **<index + F> 5 5 2** when, e.g. in the main menu, you can start the rinsing process immediately.

- The pump rinses until you stop the process with **RUN** or **3**.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.5.6 Dosing pump

If you wish to dispense for a certain time, press function number **6**. If you press **5 6** in the *Auxiliary functions* menu you are immediately shown the selection menu.

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameter mask. If you press **<index + F> 5 6 1** when, e.g. in the main menu, you are shown it immediately.

1. Select the sample changer: "ST20 1" or "ST20 2".
2. Enter the time [s] during which the pump attached to the sample changer should perform dispensing.
The specified time is an empirical value.

If you confirm the entry with **RUN**, you can start dispensing.

Start: press **2** or **RUN**.

- The pump dispenses for the specified time.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.5.7 Dosing pump manual

If you wish to interrupt a dispensing operation manually, press function number **7**. If you press **5 7** in the *Auxiliary functions* menu you are immediately shown the selection menu.

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

- 1. Select the sample changer: "ST20 1" or "ST20 2".

If you confirm the selection with **RUN**, you can start dispensing.

Start: press **2** or **RUN**.

If you press **<index + F> 5 7 2** when, e.g. in the main menu, you can start the dispensing process immediately.

- The pump dispenses until you stop the process with **RUN** or **3**.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.5.8 Dispenser

If you have attached a dispenser to the "DISPENSER" output of the sample changer, press function number **8**. If you press **5 8** in the *Auxiliary functions* menu you are immediately shown the selection menu.

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

- 1. Select the sample changer: "ST20 1" or "ST20 2".

If you confirm the selection with **RUN**, you can start dispensing.

Start: press **2** or **RUN**.

If you press **<index + F> 5 8 2** when, e.g. in the main menu, you can start the dispensing process immediately.

- The relay at the "DISPENSER" output closes for 0.5 s, thereby starting the dispensing.

You can repeat the process with **RUN**.

- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.6 Auxiliary instrument

With the aid of this function you can control a pump, a dispenser, an electromagnetic valve or a relay attached to an auxiliary output of the titrator. There is no need to install these auxiliary instruments if you do not include them in a method.

If you press function number **6** (in one of the other menus **<index + F>**, then **6**), you are shown the following menu:

- 1 Time-controlled
- 2 Manual

4.6.1 Time-controlled

If the auxiliary instrument has to perform its function at a certain time, press function number **1**. If you press **6 1** in the *Auxiliary functions* menu, you are immediately shown the selection menu:

- 1 Modify
- 2 Start

Modify: press **1**.

You are shown the parameters whose value or name you can change. If you press, **<index + F> 6 1 1** when, e.g. in the main menu, you are shown the parameter mask immediately.

1. Select the auxiliary output from the selection menu.
2. Enter the time [s]. This is the time during which the voltage of 24 V should be applied to the auxiliary output.

With the aid of a pump or dispenser you can dispense exact volumes if you know the dispensing rate for the particular solvent.

If you confirm one of the parameters with **RUN**, you can activate the instrument.

Start: press **2** or **RUN**.

- At the auxiliary output the voltage of 24 V is applied for the specified time.

You can repeat the operation with **RUN**.

- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.6.2 Manual

If you wish to stop the function of the auxiliary unit manually, press function number **2**. If you press **6 2** in the *Auxiliary functions* menu you are immediately shown the selection menu:

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

1. Select the auxiliary output from the selection menu.

If you confirm the entry with **RUN**, you can activate the instrument.

Start: press **2** or **RUN**.

If you know what auxiliary output is stored, you can, e.g. when in the main menu immediately start the function of the auxiliary instrument with **<index + F> 6 2 2**.

- The voltage of 24 V remains applied to the auxiliary output until you stop the operation with **RUN** or **3**.
- To obtain the *Auxiliary functions* menu again, press **<index + F>**.

4.7 Calibration of the temperature sensors

With the aid of this function you can calibrate your Pt100 or Pt1000 sensors to be certain that the measured temperature values are correct.

Note: Section 11.1.7 describes how to set the temperature options for use of either the Pt100 or Pt1000 sensors.

If you press function number **7** (in one of the other menus **<index + F>**, then **7**), you are shown the following menu:

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

1. Select the temperature input from the selection menu.

If you confirm the selection with **RUN**, you can calibrate the temperature sensor.

Start: press **2** or **RUN**. You are shown the following:

Immerse sensor in ice water

Continue

Terminate

- Immerse the sensor in ice water (we recommend to use a Dewar vessel!).
- Wait for some time, so that the sensor can cool to 0 °C.
- Then confirm "Continue" with **RUN**.

In the selection menu "3 Stop" appears. The temperature sensor is calibrated as soon as "2 Start" reappears.

Should the measured temperature value not lie within the range of -2 °C to +2 °C, the following error message will appear:

Measured value out of limits

Measured value not stored

- Confirm the message with **RUN** and first check the temperature value given by the sensor using the auxiliary function **Temperature**.

4.8 Offset adjustment of the sensor inputs

With the aid of this function and the short circuit plug included in the standard equipment, you can adjust the sensor inputs yourself. This is necessary when

- the error message "Sensor inputs not adjusted" is shown (see Note) or
- you insert a RS or temperature option yourself.

If you press function number **8** (in one of the other menus <**index + F**>, then **8**), you are shown the following menu:

- 1 Modify
- 2 Start

Modify: press **1**. You are shown the parameter mask.

1. Select the sensor input from the selection menu.

If you confirm the selection with **RUN**, you can adjust the sensor input.

Start: press **2** or **RUN**. You are shown the following:

Insert short circuit plug

Continue

Terminate

- Insert short circuit plug in the selected sensor input and confirm "Continue" with **RUN**. In the selection menu "3 Stop" appears. The sensor input is adjusted as soon as "2 Start" reappears.

Note: The error message "Sensor inputs not adjusted" will appear for adjusted sensor inputs when system data have been deleted from the user data memory.

- Measure:**
- Switch titrator off and on again: the system data will be restored.
 - Adjust sensor inputs.

Contents	Page
5. DOCUMENTATION	5-3
5.1 Print	5-3
5.1.1 Methods	5-3
5.1.3 Installation data	5-4
5.2 Data transfer	5-5
5.2.1 Method	5-5
5.2.2 Installation data	5-6
5.3 Memory copy	5-7
5.3.1 Request by computer for a copy of data stored in titrator	5-7
5.3.2 Request by titrator for a copy of data stored in computer	5-8
5.3.3 Request by Titrator 1 for a copy of data stored in Titrator 2	5-9

5.1.2 Installation data

You can print out the following installed resources (see Section 1):

Titrants

Sensors

Temperature sensors

Auxiliary reagents

Auxiliary instruments

Auxiliary values

Titration stands

Peripherals

Miscellaneous

1. If you select **Titrants** you obtain a list of the installed titrants with the corresponding parameters.
2. If you select **Sensors** you obtain a list of the installed sensors with the corresponding parameters.
3. If you select **Temperature sensors** you obtain a list of the installed Pt sensors with the corresponding parameters.
4. If you select **Auxiliary reagents** you obtain a list of the installed auxiliary reagents with the corresponding parameters.
5. If you select **Auxiliary instruments** you obtain a list of the installed auxiliary instruments with the corresponding parameters.
6. If you select **Auxiliary values** you obtain a list of the stored auxiliary values with the corresponding parameters.
7. If you select **Titration stands** you obtain a list of the titration stands with the corresponding parameters.
8. If you select **Peripherals** you obtain a list of all peripheral units with the corresponding parameters.
9. If you select **Miscellaneous** you obtain a list of all submenus with the corresponding parameter values, names or text.

5.2 Data transfer

You can use this menu to transfer the methods and installation data which you have stored in the computer to the titrator. For this, you must have

- attached the computer to the computer interface,
- installed it under **Peripherals** in the Installation menu, and
- loaded the DLWin software or your own program.

Computer:

- Switch on the computer and start "DLWin".

Titrator:

- Switch on the titrator, press **<Index + D>** and select **Data transfer**. The following menu appears:

```
Data transfer
Method
Installation data
```

It is also possible to transfer the methods and installation data you have stored in Titrator 1 to Titrator 2. For this, you must have

- installed a computer in the Installation menu for both instruments, and
- connected the instruments via their computer interfaces using the printer cables interconnected by a null modem.
- Switch on both titrators and select **Data transfer** in the second titrator (see above).

Note: A null modem is not included in the standard equipment. The required pin assignment of the connector is thus shown in Section 11.2.11.

5.2.1 Method

You would like to transfer a method to the titrator.

- Confirm **Method** with **RUN**: the method identification mask appears.
- Enter the ID of the desired method and confirm with **RUN**.

The message "Data transmission active" appears. The method is added as a user method, the method identification mask then reappears.

Caution: A method with the same ID will be overwritten!

Error messages:

1. If the method is not stored in the computer, the following message appears:

```
Computer: Sequence error  
Order code  
Error code    (E033 / E037 / E054) *
```

– Confirm the message with **RUN**.

* Error code E033: User data memory full

Error code E037: Working data memory full (too many resources in a block)

Error code E054: **RESET** has been pressed on the titrator which receives the data.

2. If a method with the same ID is active or is stored in the method list (ANALYSIS), the following error message appears on the computer:

```
Command not possible  
Terminate all actions.  
Delete all methods in  
method list (ANALYSIS)
```

– Confirm the error message with **RUN**,

– terminate the method with **RESET** and/or press <**Index + A**> (<Index + B>) and delete the method from the appropriate list.

– Return to the documentation menu with <**Index + D**>.

5.2.2 Installation data

For example, you would like to transfer the list of titrants to the titrator.

- Confirm **Installation data** with **RUN**: the list of all resources appears.
- Confirm **Titrants** with **RUN**.

The message "Data transmission active" appears.

- Titrants stored in the titrator will be overwritten if their **names** and **concentrations** are identical to those transferred. All other titrants remain stored.
- Sensors, auxiliary reagents and auxiliary instruments stored in the titrator will be overwritten if their **names** are identical to those transferred.
- All other resources (temperature sensors, auxiliary values, titration stands, peripherals, miscellaneous) will be overwritten.

Error message

If the installation data are not stored in the computer, the message "Computer: Sequence error" appears (see above: Error message 1).

5.3 Memory copy

You can use this menu

- to allow the computer to request a copy of all user methods and installation data stored in the titrator, or
- to request a copy of all methods and installation data stored in the computer by the titrator, or
- to request a copy of all methods and installation data stored in the first titrator by a second titrator.

You do not require an RS option for this.

You must have

- attached the computer to the **printer interface** of the titrator, and
- loaded the DLWin software or your own program.

Note: No data for the peripherals will be copied!

5.3.1 Request by computer for a copy of data stored in the titrator

Computer:

- Plug computer connection cable into the printer port of the titrator.
- Switch on the computer and start "DLWin".

Titrator:

- Switch on the titrator, press **<Index + D>** and select **Memory copy**. The following menu appears:

Memory copy

Prepare

Request

- Confirm **Prepare** with **RUN**. This command causes data to be transmitted via the printer interface.

Computer:

- Select **Backup** in the main menu.
- Select **Request memory copy** in the backup menu: the data are copied.

Titrator:

"Data transmission active" appears in the display. As soon as the copying operation is at an end, the main menu appears in the display indicating that data transmission will now once again be routed across the computer interface.

- Set up original cabling configuration!

Note: All errors which can appear during the storage operation are listed in the Operating Instructions enclosed with the RS option.

5.3.2 Request by titrator for a copy of data stored in the computer

Computer:

- Plug computer connection cable into the printer port of the titrator.
- Switch on the computer and start "DLWin".

Titrator

- Switch on the titrator, press **<Index + D>** and select **Memory copy**. The following menu appears:

Memory copy

Prepare

Request

- Confirm **Request** with **RUN**: The following menu appears:

Overwrite memory?

No

Yes

No: The procedure will be terminated.

Yes: The user methods and installation data will be copied. The display shows "Data transmission active".

Caution: All methods with the same **ID** will be overwritten!

The stored titrants will be overwritten if their **names** and **concentrations** are identical to those copied!

The stored sensors, auxiliary reagents and auxiliary instruments will be overwritten if their **names** are identical to those copied.

All other resources (temperature sensors, auxiliary values, titration stands, miscellaneous) will be overwritten.

As soon as the copying operation is at an end, the main menu appears in the display indicating that data transmission will now once again be routed across the computer interface.

- Set up original cabling configuration!

Error messages:

1. If you select **Memory copy** while methods and/or auxiliary functions are active and/or methods are stored in the method list under ANALYSIS, the following appears:

```
Command not possible  
Terminate all actions.  
Delete all methods in  
method list (ANALYSIS)
```

- Confirm the error message with **RUN**,
 - terminate all activities with **RESET**, and/or press <Index + A> (<Index + B>) and delete the methods from the appropriate list.
 - Return to the documentation menu with <Index + D>.
2. When the user data memory is full, the computer displays an appropriate error message.

5.3.3 Request by Titrator 1 for a copy of data stored in Titrator 2

A memory copy from one titrator to the other is possible only if you interconnect the two printer cables using a null modem (see Note on page 5-5).

- Switch on both titrators.

Titration 2:

- Press <Index + D> and select **Memory copy**. In the menu

```
Memory copy  
Prepare  
Request
```

- confirm **Prepare** with **RUN**: This command causes data to be transmitted via the printer interface.

Titration 1:

- press <Index + D> and select **Memory copy**. In the menu

```
Memory copy  
Prepare  
Request
```

- confirm **Request** with **RUN**.

The following menu appears:

Overwrite memory?

No

Yes

No: The procedure will be terminated.

Yes: The user methods and installation data will be copied. The display shows "Data transmission active".

Caution: All methods with the same **ID** will be overwritten!

The stored titrants will be overwritten if their **names** and **concentrations** are identical to those copied!

The stored sensors, auxiliary reagents and auxiliary instruments will be overwritten if their **names** are identical to those copied.

All other resources (temperature sensors, auxiliary values, titration stands, miscellaneous) will be overwritten.

As soon as the copying operation is at an end, the main menu appears in the display indicating that data transmission will now once again be routed across the computer interface.

- Set up original cabling configuration!

Error messages:

1. If you select **Memory copy** (with Titrator 1 or 2) while methods and/or auxiliary functions are active and/or methods are stored in the method list under ANALYSIS, the following appears:

Command not possible

Terminate all actions.

Delete all methods in
method list (ANALYSIS)

- Confirm the error message with **RUN**,
- terminate all activities with **RESET** and/or press <Index + A> (<Index + B>) and delete the methods from the appropriate list.
- Return to the documentation menu with <Index + D>.

2. If errors appear during the storage operation, Titrator 2 shows the following message:

Computer: Sequence error

Order code

Error code (E033 / E037 / E054) *

– Confirm the message with **RUN**.

* Error code E033: User data memory full

Error code E037: Working data memory full (too many resources in a block)

Error code E054: **RESET** has been pressed on the titrator which receives the data.

Contents	Page
6. USER LEVEL	6-3

6. USER LEVEL

In the Installation menu under *Routine level* you have blocked the menus that should be inaccessible to employees (see Section 1.9.6). With the aid of this menu you can implement this selection.

You are shown the following parameter mask:

User level Expert

– Press **SEL**: → Routine.

If you quit this menu with **EXIT**, the menus you blocked under *Routine level* are now no longer accessible or their access is limited.

Only the accessible menus appear in the display under MAIN MENU; under the Editor menu, the METTLER and user methods are accessible for a printout only; under the Analysis menu, the submenu *Modify method* is missing.

The USER LEVEL menu is no longer visible.

To redisplay it:

– position the selector bar on the title line **MAIN MENU** and enter **E X P**.

All menus reappear on the display.

If you wish to switch over to **Expert**,

– select **USER LEVEL** and press **SEL**.

If you wish to make modifications in one of the menus but do not switch from **routine level**,

– select the menu and modify parameters in the submenu.

As soon as you confirm the title line of the menu (e.g. Installation or Editor) or press **<Index + M>**, only the accessible menus appear in the main menu: The blocked menus are opened only temporarily.

Contents	Page
7. Remote control	7-3
REMOTE CONTROL (menu)	7-3
7.1 Communication between titrator and computer	7-5
7.1.1 Overview	7-5
7.1.2 Introduction	7-5
7.1.2.1 LIMS tasks	7-5
7.1.2.2 Administration of instrument-specific data	7-6
7.1.2.3 Backup of instrument-specific data	7-7
7.1.2.4 Control of sequences (automation)	7-7
7.2 Configuration of the terminal (DEC VT340)	7-9

7. Remote control

This section describes the REMOTE CONTROL menu, provides an overview of the communication between titrator and computer and explains the configuration of an attached terminal.

REMOTE CONTROL (menu)

You can use this menu for exclusive control of the titrator by a computer, in other words you can neither enter data in the titrator nor initiate activities. For this, you must have

- installed the computer in the installation menu under **Peripherals**, and
- loaded the DLWin software or your own program.

Note: You will find further details in the Operating Instructions "RS232C Interface Description" enclosed with the RS option.

Computer:

- Switch on the computer and start "DLWin".

Titrator:

- Confirm REMOTE CONTROL in the main menu with **RUN** and
- select **On** with **SEL**.
- Quit the mask with **EXIT**. The following "MAIN MENU" appears:

```
MAIN MENU
ANALYSIS A
ANALYSIS B*          (* appears only with the DL77)
REMOTE CONTROL
```

Note: If, e.g. auxiliary functions are active or methods are entered in the method list of the Analysis menu, the following error message will be shown:

```
Command not possible
Terminate all actions.
Delete all methods in
method list (ANALYSIS)
```

- Confirm the error message with **RUN**,
- terminate all activities with **RESET** and/or press **<Index + A>** (**<Index + B>**) and delete the methods from the appropriate list.
- Press **<Index + M>** and again select REMOTE CONTROL.

To follow the entries and commands of the computer on the titrator display, you can use the following keys and key combinations:

- Arrow keys
- RUN
- EXIT
- Index + M → Main menu
- Index + A → Method list of Analysis menu A
- Index + B → Method list of Analysis menu B
- Index + S → Sample data list
- Index + T → Display of the active function of a current method

To switch off the remote control when you have finished,

- select **Off** in the REMOTE CONTROL menu.

7.1 Communication between titrator and computer

7.1.1 Overview

If you have attached and installed a computer, the titrator is able to receive and process data in a specific format from this external device or send data to the outside world.

You can attach all devices which fulfill the specifications described below. Possible devices include any type of computer, from the simple hand-held computer up to a mainframe. The titrator can thus also be integrated in relatively large systems.

In Section 7.1.2, we refer to the use of the system titrator <—> computer.

The functions available for the communication and the prerequisites for data interchange between the titrator and computer are described in the Operating Instructions enclosed with the RS option.

7.1.2 Introduction

Computers are finding increasing use in labs in the chemical industry. In association with an analytical instrument, they undertake the following tasks:

- LIMS tasks (Laboratory Information Management System)
- Administration of instrument-specific data
- Backup of instrument-specific data
- Control of sequences (automation).

7.1.2.1 LIMS tasks

At present, there exist neither standards, directions nor rules that define the tasks of a LIMS. It has become apparent, however, that the following tasks can be handled by most LIMSs:

- Generation and administration of work sheets
- Sample observation (from sampling up to archiving of the data obtained)
- Sample preparation (e.g. weighing)
- Data acquisition (online or via a keyboard)
- Archiving and administration of results and measured values
- Record generation.

Important for the titrator are sample preparation and data acquisition:

Sample data such as weight and identification must be prepared in the computer and stored in a file in a particular format.

→ The titrator accepts the sample data at a suitable time automatically.

The computer must ensure that the correct sample data are transferred in each case (see also next section).

A record or certificate must be generated by the computer. The appropriate data are needed for this.

→ The titrator transfers record data to the computer (results, measured values, etc.).

7.1.2.2 Administration of instrument-specific data

In order for you to enter sample data on the computer, you must be familiar with the given basic conditions from the method (method data).

→ The titrator sends the method data of a particular method to the computer on request.

The method data contain the parameters of the *Sample* function (see Section 2.3.2). With the method data, the same parameters as in the method data mask can be changed (see Section 3.1). With the sample data, you can enter the sample identification 2, the weight/volume and the correction factor f. To undertake these changes, you must have loaded an appropriate program on your computer.

You must manage the methods stored in the titrator from the computer:

→ The titrator sends a list of its stored methods on request from the computer.

→ The titrator sends a record of the desired method with its functions and parameters on request from the computer.

You must manage the installation data stored in the titrator using the computer:

→ The titrator sends a record of the desired installation data on request from the computer.

7.1.2.3 Backup of instrument-specific data

All instrument-specific data decisive for an analysis must be stored in a central unit. Such data can pertain to methods or installation and may be changed only in the titrator (e.g. in the EDITOR). On the one hand this meets GLP (Good Laboratory Practice) demands, on the other hand problems with different versions of a method (date/time) can be avoided if several titrators are attached to the computer.

→ It is possible to interchange methods and installation data between the computer and the titrator.

7.1.2.4 Control of sequences (automation)

A method must be started in the computer (remote control)

→ The remote control can be switched on and off in the REMOTE CONTROL menu of the titrator (status "On" or "Off").

On: The titrator is controlled exclusively by the computer. You can use the titrator only to follow the progress of the analysis (shortened menu).

Off: The Analysis working area is accessible from the computer and by you from the titrator. As long as methods are entered and processed, the working area remains reserved for the respective "Master".

With the DL77 you can start a method in working area A from the computer and at the same time one in working area B from the titrator.

→ Methods are added in the computer to the method list of the titrator. The method is started with "List once" if a method is not already running. The sequence of the methods can then be synchronized only with the computer.

→ Entries which need a method during an analysis are requested from the computer by the titrator (e.g. "Current sample").

→ The sequence status (e.g. "List processed") and the error status (e.g. "Short circuit") is sent to the computer.

The most important auxiliary functions must be initiated at the computer. These are

- Rinsing a burette
- Dispensing
- Measuring a potential (steady state)
- Stirring
- Auxiliary instrument (time-controlled)
- Entering a titer
- Entering an auxiliary value.

This results in a wide range of additional remote control possibilities.

External operations should be synchronized with the sequence of an analysis. This synchronization is explained in what follows with the aid of a few examples:

1. When you work at the computer, your attention must be drawn to any result calculated during the analysis that lies outside the limits.
 - The titrator sends an appropriate signal to the computer. The computer shows you the message corresponding to this signal.
2. In addition to the titrator, the computer controls, e.g. a robot. The task of the robot is to insert the samples in the titrator.
 - The titrator has to tell the computer when the next sample should be inserted and wait until this has happened (*Request to continue*). The computer for its part knows when the robot has inserted the sample and informs the titrator of this so that it can continue the analysis (*Allow titrator to continue*).

To allow several titrators to be attached to a low-end computer, the titrator can be so configured that it will transmit data to the computer only when this sends the appropriate inquiry (polling).

7.2 Configuration of the terminal (DEC VT340)

You have attached the terminal to the titrator and have installed it (see Section 1.8.3). To configure it:

- Switch off the titrator,
- switch on the terminal and wait until the message **VT340 OK** is displayed.
- Press the **Set-Up** key: the **SET-UP DIRECTORY** is displayed.
- a. If you have already configured the terminal for other connections, first select **Recall Factory Default Settings** to reactivate the default configuration parameters.
- b. If you have not used the terminal before, you can modify the default parameters immediately.
- Select **General Set-Up** and modify the following parameter:

Terminal mode	VT300-8bit
---------------	------------
- Select **Display Set-Up** and modify the following parameters:

Scrolling	jump
Status Display	host writable
- Select **Communications Set-Up** and modify the following parameters:

Transmit Speed	9600 *
Receive XOFF Point	512
Character Format	8 bits, even parity *

* These parameters must correspond to the entered installation data!
- Select **Keyboard Set-Up** and modify the following parameter:

Keypad mode	numeric:	if the keys of the numeric keypad (at extreme right of keypad) should be active.
	application:	if "key combinations" should replace the numbers of the numeric keypad (see following page).
- Select **Save Current Settings** to save the modified parameters.
- Press the **Set-Up** key to quit the SET-UP DIRECTORY.
- Switch on the titrator: the terminal display is now activated by the titrator.














If you have switched off both instruments,

- **first** switch on the terminal, **then** switch on the titrator.

You can use the keypad of the terminal to operate the titrator. The keys are assigned as follows:

RUN	→	Do
EXIT	→	F17
RESET	→	F20
SEL	→	Select
CE	→	Remove
HELP	→	Help
i	→	Find
↑ + ↑	→	Prev. Screen
↑ + ↓	→	Next Screen

If you have saved **application** as a parameter for the numeric keypad, the numbers are assigned to the following key combinations of the titrator:

 + M	→	0	(MAIN MENU)
 + I	→	1	(INSTALLATION menu)
 + E	→	2	(EDITOR menu)
 + A	→	3	(ANALYSIS A menu)
 + D	→	4	(DOCUMENTATION menu)
 + F	→	5	(AUXILIARY FUNCTIONS menu)
 + S	→	6	(Sample data list)
 + T	→	7	(Display of the current method)
 + B	→	8	(ANALYSIS B menu)
 + U	→	,	(Rinse burette)
 + Y	→	.	(Rinse tip)
 + L	→	PF1	(Line feed on the printer)
 + P	→	PF2	(Form feed on the printer)

Contents	Page
8. Designations – Explanations – Examples	8-3
8.1 List of designations	8-3
8.1.1 Compilation of the raw results	8-9
8.2 Use of indexes	8-10
8.2.1 Compilation of designations according to indexing forms	8-14
8.3 Functions with a condition	8-16
8.4 Evaluation procedures	8-20
8.4.1 Standard	8-20
8.4.2 Asymmetric	8-21
8.4.3 Segmented	8-22
8.4.4 Minimum/maximum	8-23
8.5 Restrictions in method development and execution	8-24
8.5.1 Maximum number of functions per method	8-24
8.5.2 Maximum number of samples	8-24
8.5.3 Maximum number of methods in the method list of the Analysis menu	8-25
8.5.4 Maximum number of equivalence points per method	8-25
8.5.5 Maximum number of measurement points per Titration function	8-25
8.5.6 Maximum number of results per method	8-25
8.5.7 How long does the titrator store data?	8-25
8.6 Examples of formulae	8-26
8.6.1 Results	8-26
8.6.2 Constants	8-27
8.6.3 Nominal content	8-28
8.6.4 Formulae for limiting the equivalence point	8-31
8.7 Examples of methods	8-32
8.8 Scheme for method design	8-36

8. Designations – Explanations – Examples

In this section you will find additional information, explanations and examples that supplement the EDITOR section.

8.1 List of designations

This list shows you the abbreviations of all designations of the parameters and of the raw results determined by the titrator and their definition. You must adhere to the upper case or lower case notation of the designations during entries, otherwise the titrator outputs an error message.

Titrant

c Nominal concentration of the titrant in mol/L (**Dispense**, **Titration**, **pH/mV-stat**, **Calculation** functions)

The actual equivalent concentration (ACTUAL value) of the titrant, the product of the nominal concentration **c** and titer **t** in mol/L, is calculated automatically by the titrator.

Sample function

U Sample volume in mL (for the **Calculation** function)

m Sample weight in g (for the **Calculation** function)

M Molar mass in g/mol (for the **Calculation** function)

z Equivalent number: number of reaction entities of the sample compared to the titrant (for the **Calculation** function)

Note: The notation **z*** of DIN standard 32 625 has not been used for the titrator for technical reasons.

f Correction factor (for the **Calculation** function)

Measure function

E Measured value in mV or in the installed unit of the sensor used

Temperature function

T Measured temperature in °C, °F or K of the Pt sensor used

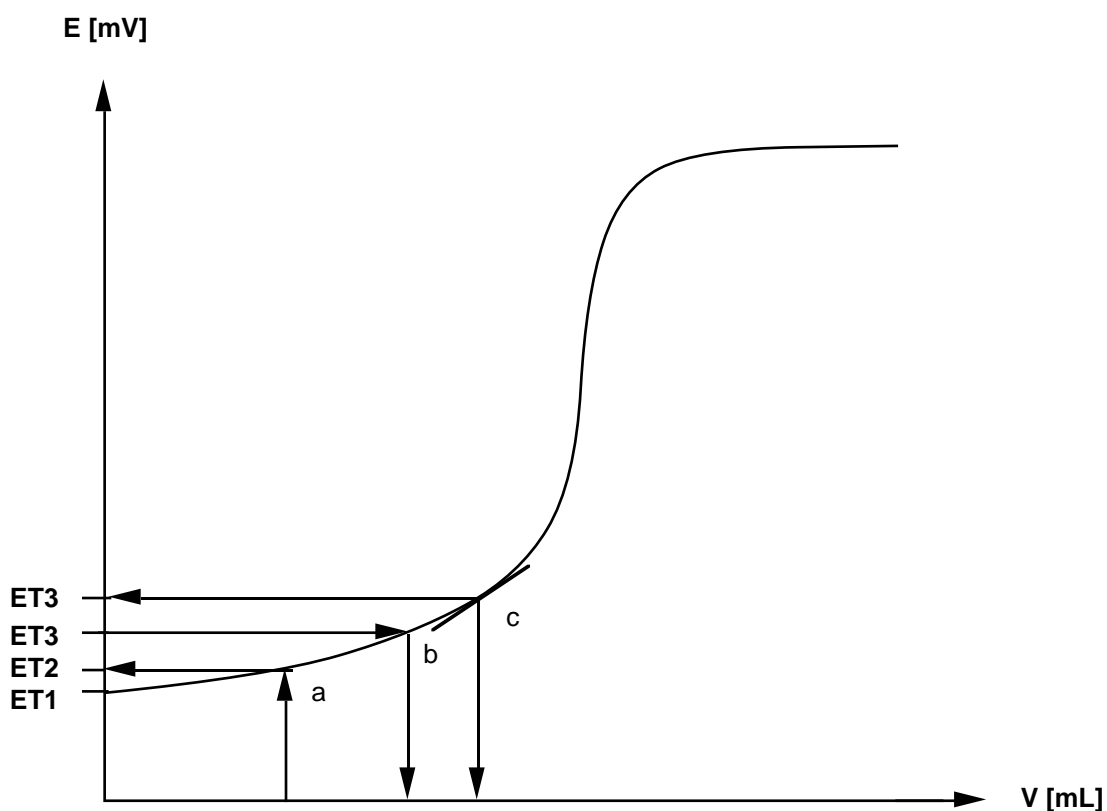
Dispense function

VDISP Total dispensed volume in mL

QDISP Total dispensed amount of substance in mmol

Titration function

- ET1** Initial potential of the measured solution before the first titrant addition in mV or in the installed unit of the sensor used
- ET2** Potential following Dose 1, Predispensing 1 or Predispensing in mV or in the installed unit of the sensor used (*Titration mode DOS, EQP, and EP*)
- ET3** Potential following Dose 2 or Predispensing 2 in mV or in the installed unit of the sensor used (*Titration mode DOS and EQP*)



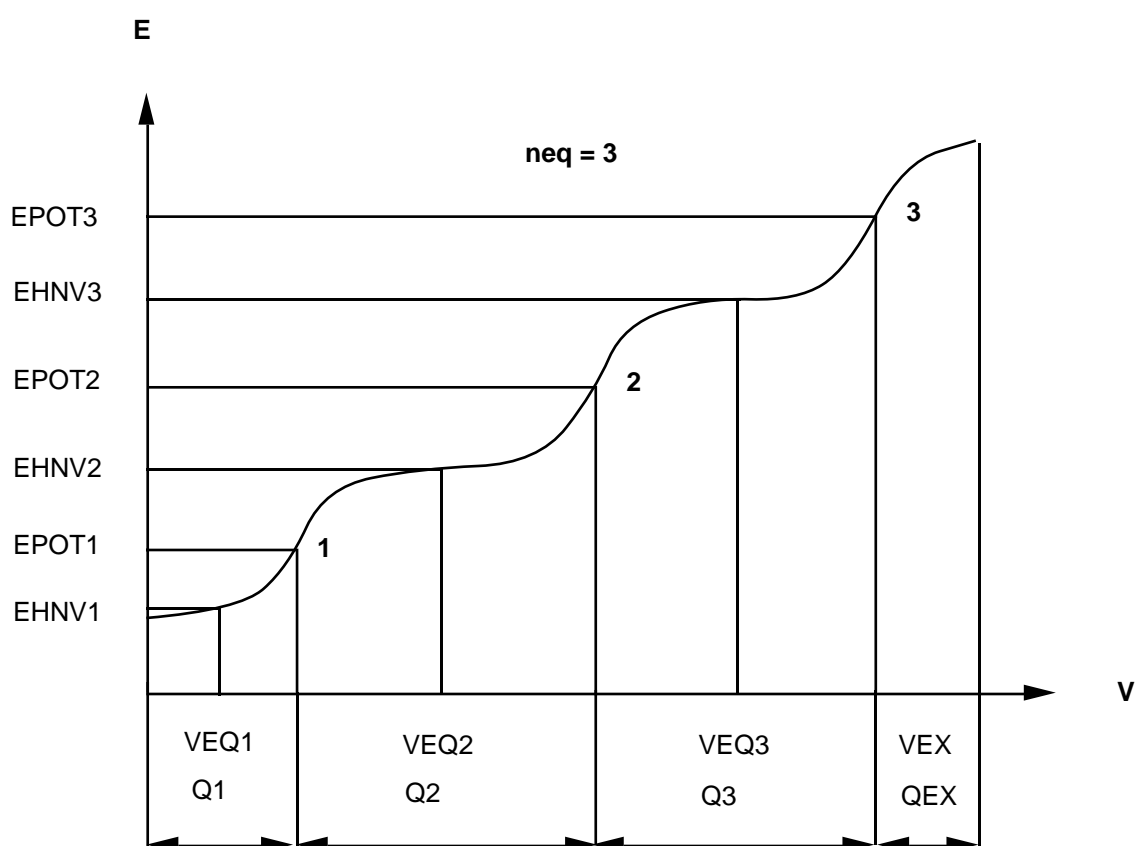
a: mL or nominal content dispensing

b: dispensing to a potential

c: dispensing to the slope of the curve

Titration function

VEQ	Titrant consumption in mL up to the equivalence or end point; the equivalence points are numbered consecutively
Q	Titrant consumption in mmol up to the equivalence or end point; the equivalence points are numbered consecutively
VEX	Calculated excess of the titrant in equivalence point or end point titrations in mL
QEX	Calculated excess of the titrant in equivalence point or end point titrations in mmol
EPOT	Calculated equivalence point potential of the equivalence or end point VEQ in mV or in the installed unit of the sensor used
EHN	Calculated half neutralization value (potential at VEQ/2) referring to the equivalence point VEQ in mV or in the installed unit of the sensor used
neq	Number of equivalence points found (applies to one Titration function)

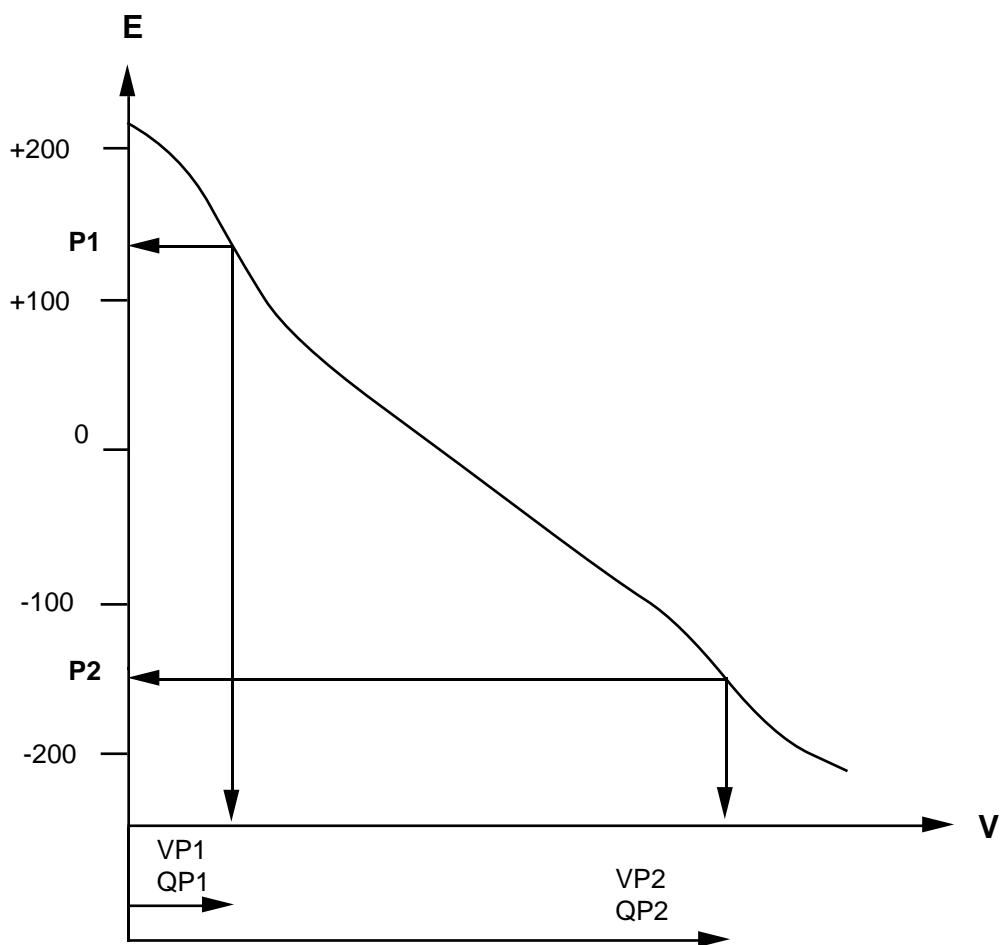


Titration function

VP1 Titrant consumption in mL up to buffer potential P1
VP2 or P2

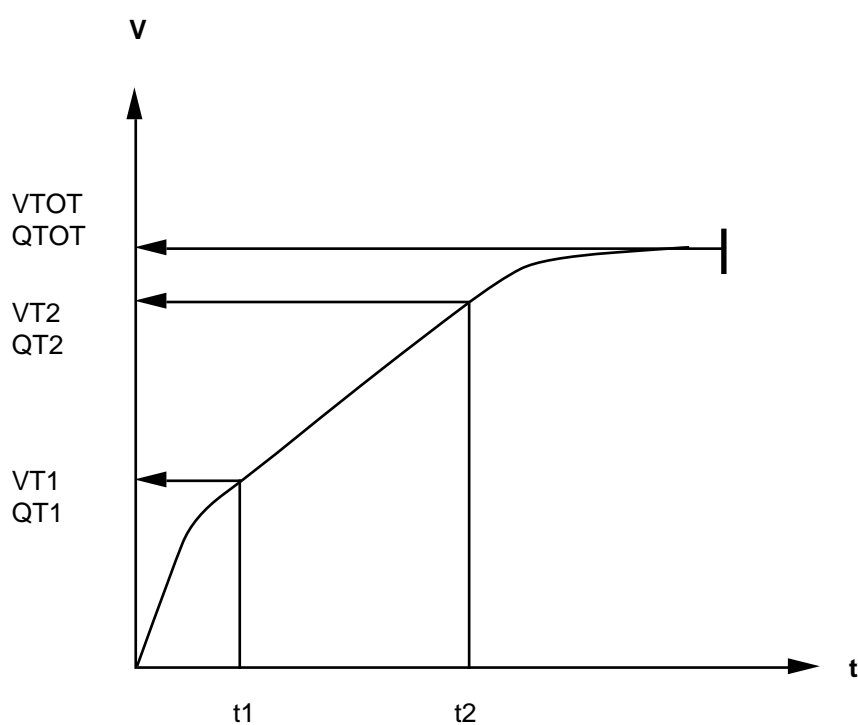
QP1 Titrant consumption in mmol up to the buffer potential P1
QP2 or P2

P1 Buffer potentials in mV or in the installed unit of the sensor used
P2



pH/mV-stat **function**

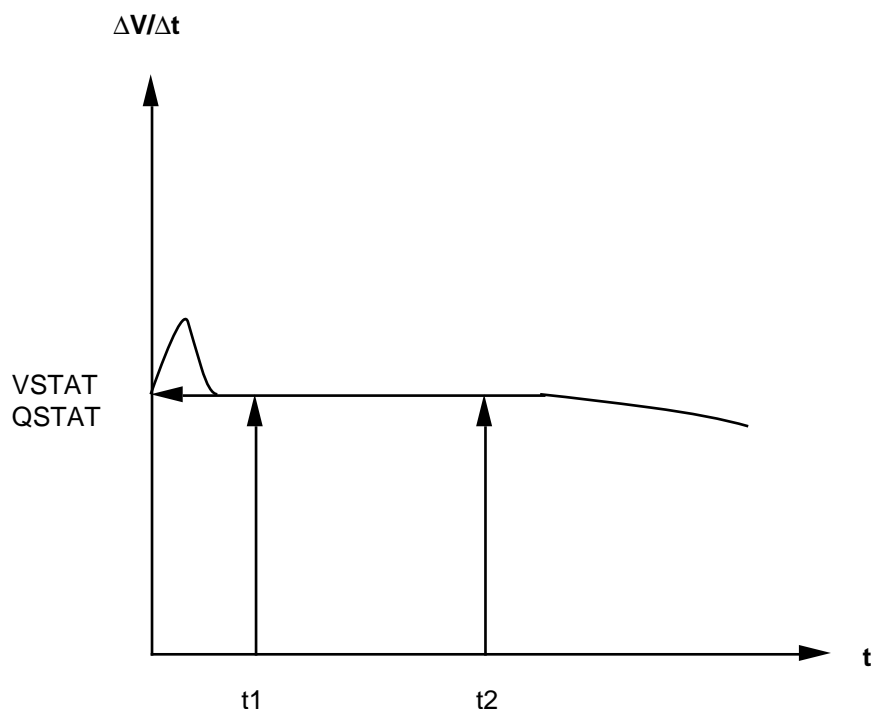
VTOT	Total dispensed volume in mL
QTOT	Total amount of substance dispensed in mmol
VT1	Titrant consumption in mL up to time limit t1 or t2
VT2	
VT	Titrant consumption in mL up to a different time limit
QT1	Titrant consumption in mmol up to time limit t1 or t2
QT2	
QT	Titrant consumption in mmol up to a different time limit
CSTAT	Correlation coefficient of the regression lines between t1 and t2 of the V – t curve



pH/mV-stat **function**

VSTAT Mean titrant consumption in mL/min within the time limits t1 and t2

QSTAT Mean titrant consumption in mmol/min within the time limits t1 and t2

Calculation **function**

R Calculated result

C Calculation constant

Titer **function**

t Titer

Auxiliary **function**

H Auxiliary value

Statistics **function**

\bar{x} Mean value

s Standard deviation

srel Relative standard deviation in %

Current method

TIME Elapsed time of a current method in seconds (see Sections 3.1.3 and 10.2.7).

8.1.1 Compilation of the raw results

Method / Function	Raw results	Printed, if "Raw results last sample" is selected in the Record function
Current method	TIME	no
MEASURE	E	yes
TEMPERATURE	T	yes
DISPENSE	VDISP, QDISP	yes
TITRATION	ET1	no
	ET2, ET3	no
	VEQ, Q	yes
	VEX, QEX	yes
	EPOT	yes
	EHNV	no
	neq	yes
	P1, P2	no
	VP1, QP1	no
	VP2, QP2	no
pH/mV-STAT	VTOT, QTOT	yes
	VT1, QT1	no
	VT2, QT2	no
	VT, QT	no
	VSTAT, QSTAT	yes
	CSTAT	no

Note: To obtain the raw results, which are not printed out by default

- assign them to the result **R**, e.g. R = QP1, R = VT1.

8.2 Use of indexes

Since functions can occur more than once within a method, and also parameters can appear repeatedly within a function, the parameter designation must be qualified by indexes.

The titrator recognizes **4** different indexing forms:

1. Parameters without index

The following designations are associated with these parameters:

c	t	m	U	M
z	f	TIME		

The parameters always refer to the current titrant, the current sample, or the current method.

2. Parameters of the form **Xi**

All parameters used without bracketed index have no meaningful connection with the method function. The following designations are associated with this indexing form:

R	C	H
---	---	---

Example: **R3** **third** result (see example under indexing form **4**)

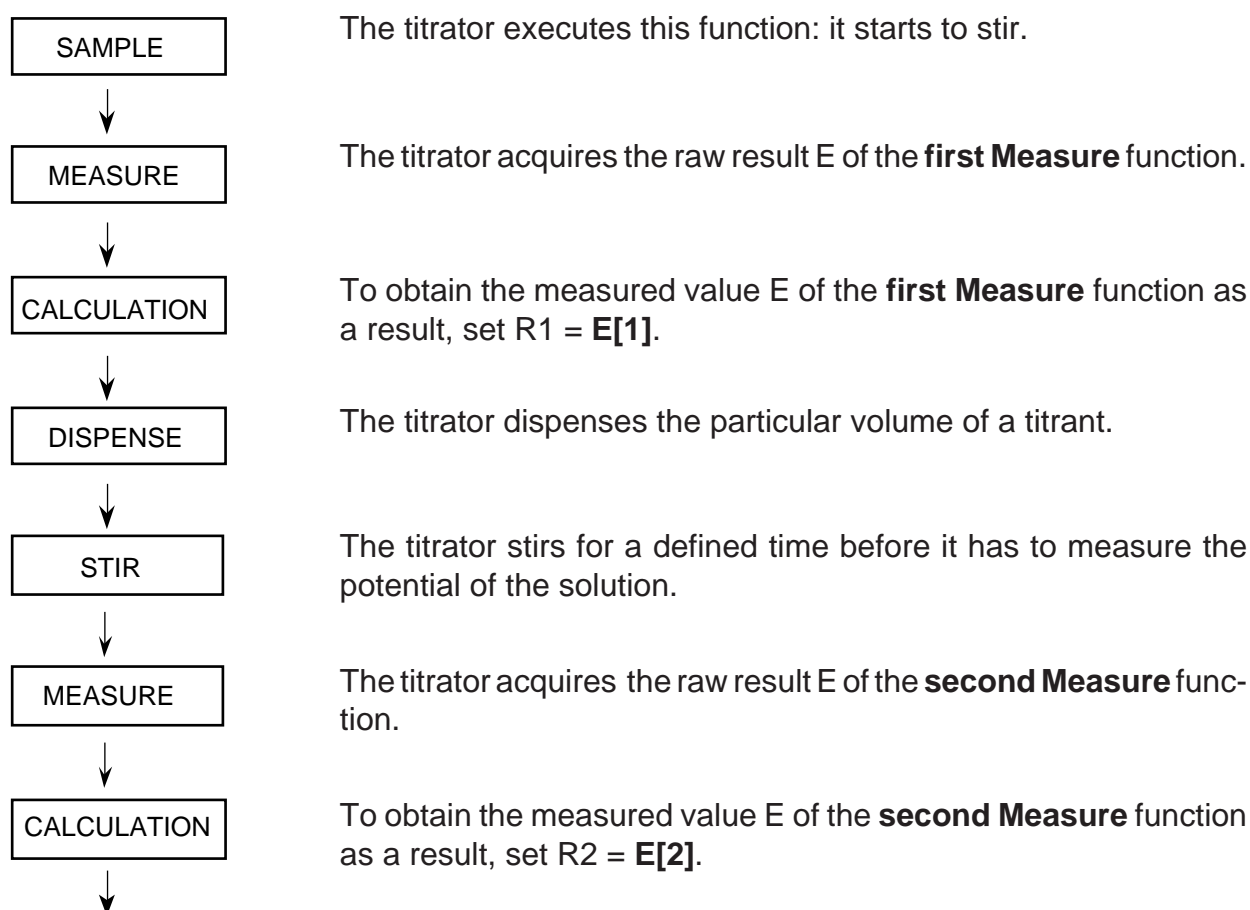
Note: **R = R1** applies, in other words the index **1** can be omitted. This also applies to the indexing forms under points 3 and 4.

3. Parameters of the form **X[j]**

Index **j** is the function counter. The following designations are associated with this indexing form:

E	T	VDISP	QDISP	ET1
ET2	ET3	VEX	QEX	neq
VP1	VP2	QP1	QP2	P1
P2	VTOT	QTOT	VT1	VT2
QT1	QT2	VSTAT	QSTAT	CSTAT
\bar{x}	s	srel		

Example: **E[2]** Measured value of the **second Measure** function (excerpt from a method)



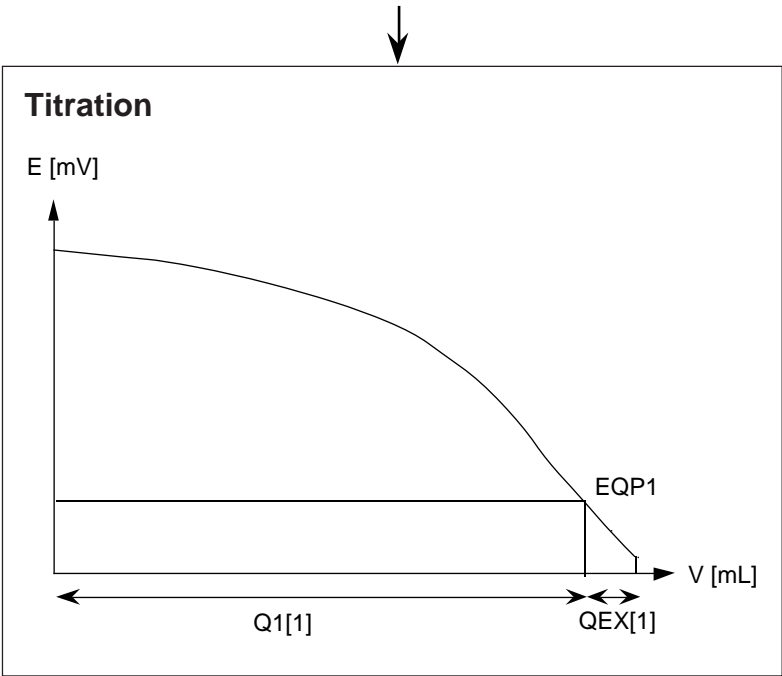
4. Parameters of the form **$X_i[j]$**

Index **i** is the number of the parameter within a function. Index **j** is the counter for the number of functions within a method. The following designations are associated with this indexing form:

Q VEQ EHNV EPOT

Example: **Q2[2]** mmol consumption up to the second equivalence point of the **second Titration** function.

Excerpt from a method: determination of HCl, CH₃COOH und NH₄Cl with NaOH
(**Two** Titration functions were selected, because for CH₃COOH and NH₄Cl, different values were defined for the equilibrium controlled measured value acquisition EQU.)



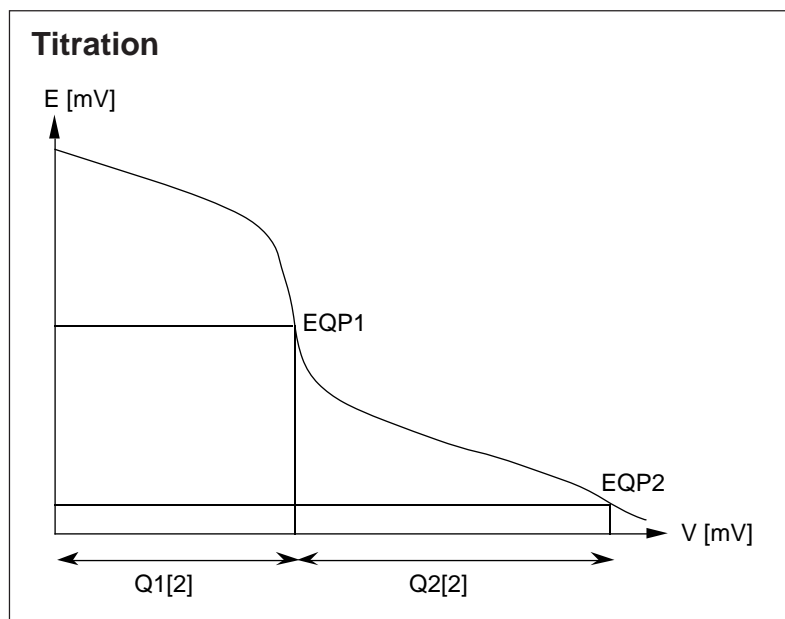
The titrator executes the **first** Ti-
tration function and titrates up to
the equivalence point of HCl. It
determines among other things
the mmol consumption Q1[1] and
the mmol excess QEX[1].

Q1[1] \equiv Q
QEX[1] \equiv QEX.

Calculation	
Result name	HCl
Formula	R1 = Q1[1]
Constant	
Result unit	mmol
Decimal places	4

In the first Calculation function,
you define the mmol consumption
for HCl.

Since you can omit index **1**, the
formula can also be R = Q.



The titrator executes the **second** Titration function. It determines among other things the mmol consumption $Q1[2]$ up to the first equivalence point (CH_3COOH), then the mmol consumption $Q2[2]$ up to the second equivalence point (NH_4Cl).



Calculation

Result name	CH_3COOH
Formula	$\mathbf{R2 = Q1[2] + QEX[1]}$
Constant	
Result unit	mmol
Decimal places	4

In the second Calculation function, you define the mmol consumption for acetic acid.

You take into account here the titrated excess of the first Titration function.



Calculation

Result name	NH_4Cl
Formula	$\mathbf{R3 = Q2[2]}$
Constant	
Result unit	mmol
Decimal places	4

In the third Calculation function, you define the mmol consumption for NH_4Cl .



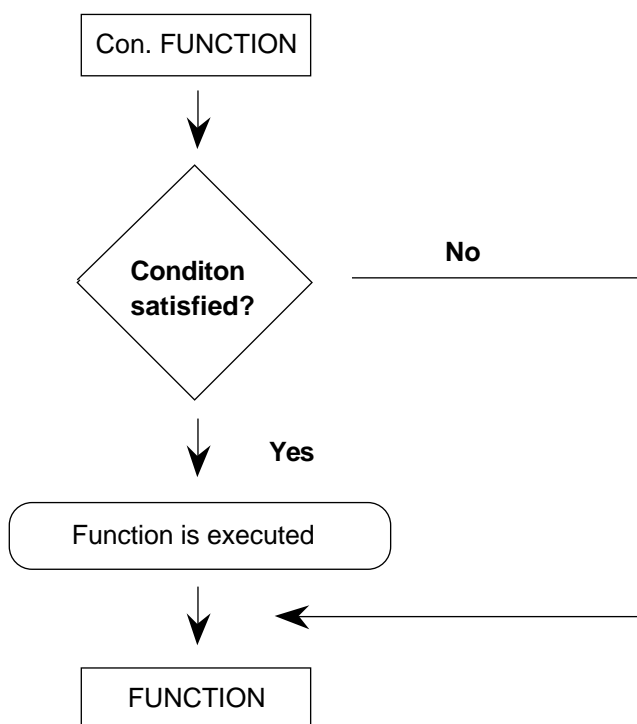
8.2.1 Compilation of designations according to indexing forms

Method / Resource/ Function	Designation [Unit]	Index	Examples
Current method	TIME [s]	--	
Titrant	c [mol/L] t		
SAMPLE	m [g]		
	U [mL]		
	M [g/mol]		
	z f		
CALCULATION	R [g] C [mL]	Xi	H1...H20
AUXILIARY VALUE	H		
TITRATION	VEQ [mL] Q [mmol] EPOT [mV, pH ...] EHNV [mV, pH ...]	Xi[j]	Q2[1] EPOT1[2]

Function	Designation	[Unit]	Index	Examples
MEASURE	E	[mV, pH ...]	X[j]	E[3]
TEMPERATURE	T	[°C, °F, K]		
DISPENSE	VDISP QDISP	[mL] [mmol]		QDISP[2]
TITRATION	ET1 ET2 ET3 VEX QEX neq VP1 QP1 VP2 QP2	[mV, pH ...] [mV, pH ...] [mV, pH ...] [mL] [mmol] [mL] [mmol] [mL] [mmol]		neq[3]
pH/mV-STAT	VTOT QTOT VT1 QT1 VT2 QT2 CSTAT VSTAT QSTAT	[mL] [mmol] [mL] [mmol] [mL] [mmol] [mL/min] [mmol/min]		QT1[2]
STATISTICS	\bar{x} s srel			s[3]

8.3 Functions with a condition

In titration praxis there are several applications that require a departure from the sequence defined in the series listing of the functions. For this purpose you can set a condition for most of the functions. If this condition is not satisfied, the function is skipped; if it is met, the function is executed. If no condition has been set, again the function is executed. You can not set a condition for **Title**, **Sample**, **Calibration**, and **Statistics**.



Examples of conditions are comparisons of raw results, results and numeric values that have been obtained **before** the conditional function.

You can define a condition (comparison of argument **arg** with a value **a** or **b**) by means of operators. **a** and **b** can be

- numeric values
- designations
- expressions*

* Example of an expression: "R1 + H5"

a. Comparison operators

$\text{arg} > a$	greater than
$\text{arg} \geq a$	greater than or equal to (\geq)
$\text{arg} = a$	equal to
$\text{arg} \leq a$	less than or equal to (\leq)
$\text{arg} < a$	less than
$a < \text{arg} < b$	in the range
$\text{arg} \neq a$	not equal to

Examples: $R1 > 4.2$ the first result should be greater than 4.2.
 $E < 7.0$ the potential should be smaller than pH 7.0.
 $R2 = H4$ the second result should equal auxiliary value 4.
 $R1 + H5 > E[2]$ the expression (first result plus auxiliary value 5) should be greater than the potential of the second *Measure* function.

Note: You will find additional examples in Section 8.6.4.

b. Logical operators

$a ! b$	AND
$a ? b$	OR

Example: $(R1 < 0.0) ? (R1 > 10)$ the first result should lie outside the range from 0.0 to 10.

Example 1: Determination of the neutralization value of wastewater: The pH value of the samples differs greatly so that we have to titrate on the one hand with NaOH and on the other with HCl. To execute the determinations with one method, we set the conditions for functions (excerpt from a method):

↓

Sample	
Number samples	10
Titration stand	Stand 1
Entry type	Fixed volume
Volume [mL]	10.0
ID1	Waste water
Molar mass M	0.0
Equivalent number z	1
Temperature sensor	Manual

↓

Measure	
Sensor	DG111-SC
Unit of meas.	As installed
ΔE [mV]	0.5
Δt [s]	2.0
t(min) mode	Fix
t(min) [s]	20
t(max) [s]	300

Through this function, the titrator acquires the measured value **E(pH)**.

↓

Titration	
Titrant	NaOH
Concentration [mol/L]	0.1
Sensor	DG111-SC
Unit of meas.	As installed
Titration mode	EP
Titration addition	Dynamic
$\Delta E(\text{set})$ [mV]	8.0
$\Delta V(\text{min})$ [mL]	0.02
$\Delta V(\text{max})$ [mL]	0.1
ΔE [mV]	0.5
Δt [s]	0.5
t(min) [s]	2.0
t(max) [s]	20.0
Delay [s]	15
End point mode	EPA
Potential [mV, pH, ...]	7.0
Tendency	Positive
Maximum volume [mL]	40.0
Condition	Yes
Condition	$1 < E < 7$

↓

The titrator executes this Titration function only if the measured value E determined previously is in the pH range 1 - 7.

Calculation

Result name Acidity
 Formula $R = Q * C/U$
 Constant $C = 1000$
 Result unit mmol/L
 Decimal places 2
 Condition **Yes**
 Condition $1 < E < 7$

The titrator calculates the neutralization value through the mmol consumption of base

**Titration**

Titrant HCl
 Concentration [mol/L] 0.1
 Sensor DG111-SC
 Unit of meas. As installed
 Titration mode EP
 Titrant addition Dynamic
 $\Delta E(\text{set})$ [mV] 8.0
 $\Delta V(\text{min})$ [mL] 0.02
 $\Delta V(\text{max})$ [mL] 0.1
 ΔE [mV] 0.5
 Δt [s] 0.5
 $t(\text{min})$ [s] 2.0
 $t(\text{max})$ [s] 20.0
 Delay [s] 15
 End point mode EPA
 Potential [mV, pH, ...] 7.0
 Tendency Negative
 Maximum volume [mL] 40.0
 Condition **Yes**
 Condition $7 < E < 14$

The titrator executes this Titration function only if the measured value E determined previously is in the pH range 7 - 14.

**Calculation**

Result name Basicity
 Formula $R2 = Q[2] * C2/U$
 Constant $C2 = 1000$
 Result unit mmol/L
 Decimal places 2
 Condition **Yes**
 Condition $7 < E < 14$

The titrator calculates the neutralization value through the mmol consumption of acid.

Example 2: In METTLER method M002, **Titer** is a conditional function:

Titer

Titrant NaOH
 Concentration [mol/L] 0.1
 Formula $t =$ \bar{x}
 Condition **Yes**
 Condition $0.9 < \bar{x} < 1.1$

The titrator enters the calculated mean value \bar{x} of the Statistics function in the installation data of the titrant only if it lies in the range 0.9 - 1.1.

8.4 Evaluation procedures

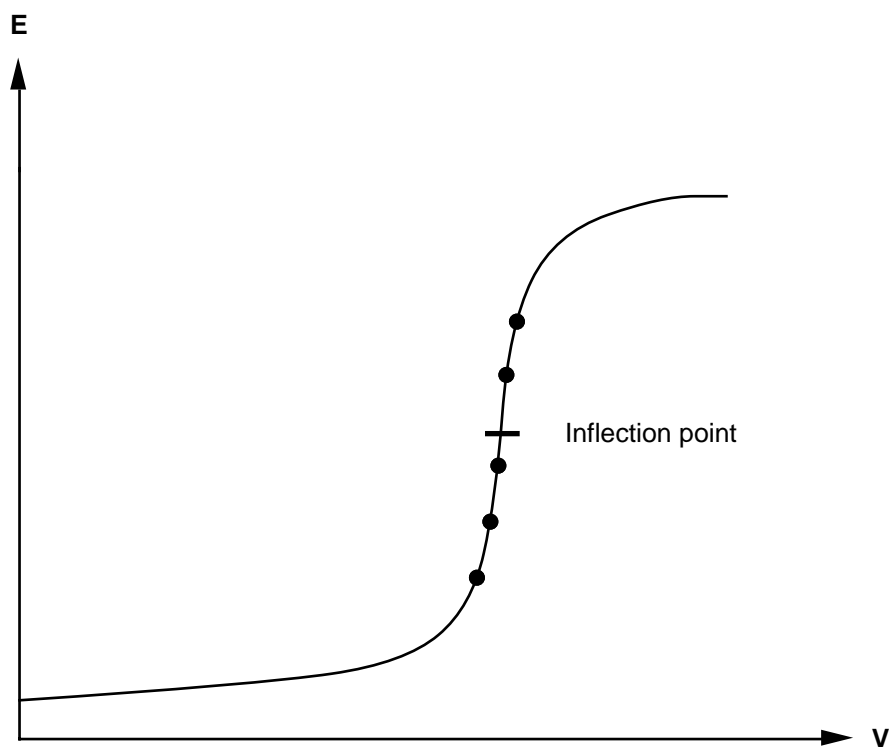
The titrator has various procedures to determine the equivalence points of a titration curve accurately:

- Standard
- Asymmetric
- Segmented
- Minimum/maximum

8.4.1 Standard

You use this procedure for all S-shaped titration curves. The evaluation is effected with the aid of an iterative procedure (nonlinear regression) [1]. The titration of a strong acid with a strong base is used as a mathematical model. The determined equivalence point in this model always lies in the vicinity of the inflection point.

At least five measurement points around the inflection point are used for the evaluation. In addition the slope from measurement point to measurement point must increase or decrease. If this condition is not met, this iterative procedure can not be employed. In such cases the determination relies on interpolation of the inflection point of the titration curve. You will be given an appropriate warning in the result record.



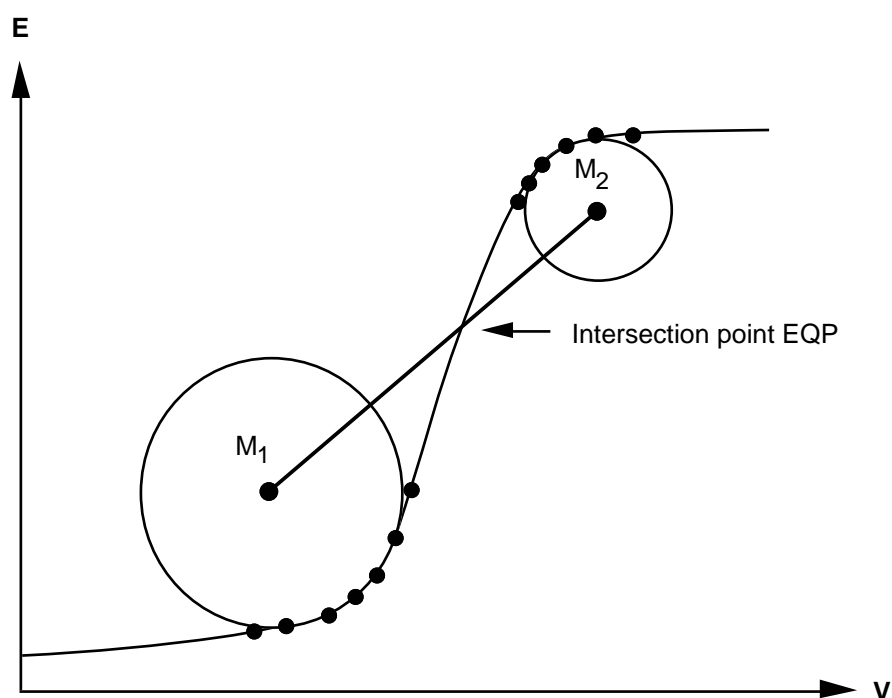
8.4.2 Asymmetric

With markedly asymmetric curves the standard procedure can lead to a systematic error. The difference between the true equivalence point and the inflection point can then be greater than the precision normally achievable. For such cases the titrator is equipped with an evaluation method following Tubbs [2].

This empirical approximation method is an old established procedure for the evaluation of asymmetric titration curves recorded in an analog fashion. It can also be used for digital determinations of titration curves [3]. The result of the Tubbs evaluation is closer than the inflection point to the true equivalence point.

The evaluation procedure is based on the following considerations:

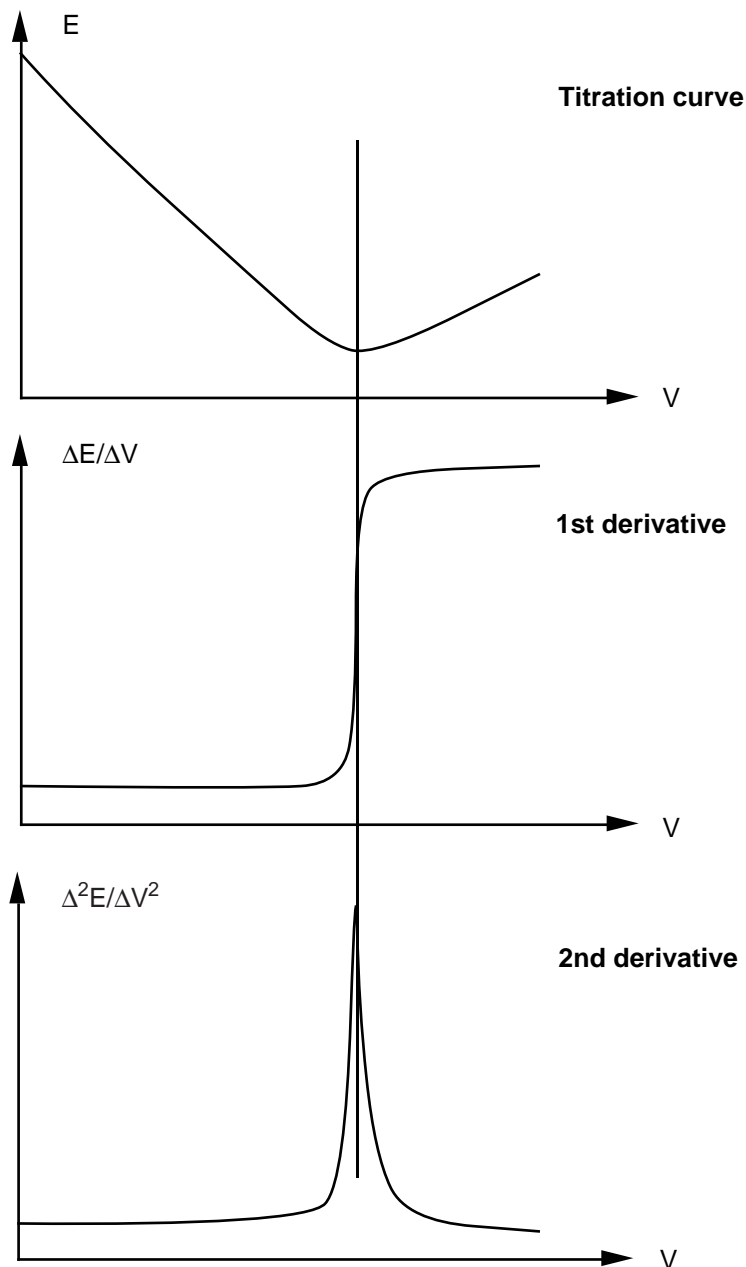
Both branches of the titration curve possess a circle of curvature with minimum radius that can be drawn in. The ratio of the two radii is determined by the asymmetry of the curve. The intersection point of the straight line connecting the midpoints M_1 and M_2 of the circles with the titration curve represents the sought-after equivalence point. Theoretical considerations show that the true equivalence point with asymmetric titration curves is always between the inflection point and that branch of the titration curve with the greater curvature (the smaller circle of curvature). The result of the Tubbs evaluation approaches the true equivalence point very closely when the course of the titration curve is regular and allows calculation of the two branches.



For the evaluation at least 6 measured points on either side of the inflection point in the region of greatest curvature are needed. If the shape of titration curve does not allow calculation of the circles of curvature, the titrator calculates the equivalence point according to the standard procedure. You will be given an appropriate warning in the result record if this is the case.

8.4.3 Segmented

Various indication methods (e.g. photometry, conductometry and amperometry) generate titration curves with linear or approximated linear sections (segmented curves). The titrator also evaluates such curves for you.



The evaluation procedure used is based on the following consideration:

The first derivative of a segmented curve displays the typical shape of an S-shaped curve whose inflection point represents a good approximation of the equivalence point.

The evaluation of segmented curves is performed with the **Standard** procedure but the calculated data of the first derivative are used rather than the data points of the titration curve.

The equivalence point is thus recognized not with the aid of the calculated first derivative but by using the calculated second derivative. The threshold for the equivalence point recognition also refers to data of the second derivative.

The individual sections do not need to be exactly linear. Decisive for an exact determination of the equivalence point is the presence of a distinct break between the individual sections of the titration curve.

8.4.4 Minimum/maximum

The result of this evaluation is the calculated minimum (maximum) from the measurement points of the titration. The classic example of a titration curve with a minimum is the determination of surfactants using photometric indication.

The minimum (maximum) is calculated by a polynomial approximation of the titration curve in the region of the minimum (maximum). The equivalence point is determined directly from the data of the titration curve.

[1] K. Waldmeier und W. Rellstab, Fres.Z.Anal.Chem., 264, 337, (1973)

[2] C.F. Tubbs, Anal. Chem., 26, 1670 (1954)

[3] S. Ebel, E. Glaser, R. Kantelberg und B. Reyer, Fres. Z. Anal. Chem., 312, 604 (1982)

8.5 Restrictions in method development and execution

8.5.1 Maximum number of functions per method

The maximum **possible** number for a particular function that you can add to a method is listed in the following Table. The number depends on the method size in each case: for example, you can **not** assemble the total of these functions to form a method as the main memory of the titrator is too small.

Function	Maximum number / method
Title	1
Sample	4
Stir	6
Measure	10
Temperature	10
Instruction	10
Dispense	6
Pump	6
Rinse	6
Conditioning	6
Auxiliary instrument	6
Titration	6
pH/mV-stat	2
Calculation	12
Auxiliary value	20
Titer	2
Calibration	2
Statistics	6
Record	8
Sync	20

8.5.2 Maximum number of samples

DL77: The sample data for a maximum of **600** samples can be entered in the method list of Analysis menu A as well as in that of Analysis menu B.
You can enter **60** sample data per method!

DL70ES: The sample data for a maximum of **600** samples can be entered in the method list of the Analysis menu.
You can enter **60** sample data per method!

DL67: This titrator can store the sample data of maximum **60** samples.

8.5.3 Maximum number of methods in the method list of the Analysis menu

DL77: A maximum of **10** methods can be entered in the method list of Analysis menu A as well as in that of Analysis menu B.

DL70ES: A maximum of **10** methods can be entered in the method list of the Analysis menu.

DL67: **One** method can be entered.

8.5.4 Maximum number of equivalence points per method

The titrator can determine maximum **16** equivalence points per method (titration mode EQP) distributed among one or several *Titration* functions. (If the method has, for example, several *Sample* functions, a maximum of 16 equivalence points will be determined, distributed among these *Sample* functions. Should more than 16 equivalence points be present, the rest will not be determined. The method, however, will be continued.

8.5.5 Maximum number of measurement points per Titration function

The titrator can store **300** measurement points per *Titration* function; it then terminates the function.

8.5.6 Maximum number of results per method

The titrator can store **180** results per method; it then terminates the method.

Example: If you titrate **30** samples with a method, the titrator can store **6** results for each individual sample.

8.5.7 How long does the titrator store data?

1. Measured values of the *Titration* or *pH/mV-stat* function

The titrator stores the measured values until the next *Titration* or *pH/mV-stat* function.

2. Raw results

The titrator stores all raw results up to the titration of the next sample within a loop. If the method has several *Sample* functions, the raw results of the sample titrated most recently within the loop will be stored.

3. Results

The titrator stores all results up to the start of the next method.

8.6 Examples of formulae

8.6.1 Results

$$R = Q * C/m \text{ (standard formula)}$$

$$C = f(M, z, \text{unit})$$

Content of a sample with weighing:
%, – ppm, – mg/g, – TAN [mg KOH/g], –
mol/kg, – mmol/g

$$R = Q * C/U \text{ (standard formula)}$$

$$C = f(M, z, \text{unit});$$

Content of a sample solution
g/L, – ppm, – % [g/mL], – mg/L, – g/100mL,
mol/L, – mmol/L

$$R = m/(VEQ * c * C)$$

Titer determined using primary standard

$$R = U/(VEQ * c * C)$$

Titer determined with volumetric solution

$$R = Q * C$$

Content per sample ($m = 1$)

$$R = Q$$

mmol consumption as result

$$R = VEQ$$

mL consumption as result

$$R = VEQ/m$$

mL/g as result

$$R = (QDISP - Q) * C/m$$

Back titration:
QDISP: dispensed amount of substance in mmol
of the *Dispense* function
Q: mmol consumption up to the equivalence point
or end point of the *Titration* function (titration
mode EQP or EP).

$$R = (Q - H_j) * C/m$$

Solvent blank value incorporated in the calculation
(blank value stored as H_j)

$$R = (Q/m - H_j) * C$$

Matrix blank value incorporated in the calculation
[mmol/g] (matrix blank value stored as H_j)

$$R = ET1 [2]$$

Initial potential of the second *Titration* function as
result

$$R = E [3]$$

Measured value of the third *Measure* function as
result

$$R = pw(-E) * 1000$$

Anion concentration in mmol/L (measured by
means of an ion-selective electrode)

8.6.2 Constants

a.	<u>Weight of sample known</u>	<u>Unit</u>
	$C = M/z$	mg/g
	$C = M/(10 * z)$	%
	$C = M * 1000/z$	ppm
	$C = 1/z$	mol/kg (mmol/g)
	$C = 56.1$	TAN or TBN (mg KOH/g)
	$C = M/(1000 * z)$	titer
b.	<u>Volume of sample known</u>	<u>Unit</u>
	$C = M/(z * 10 * d)$ where (d = density)	% [g/mL]
	$C = M * 1000/z$	mg/L
	$C = (M * 1000)/(z * d)$	ppm
	$C = 1000$	mmol/L
	$C = 1$	mol/L
	$C = M/z$	g/L
	$C = M/(10 * z)$	g/100 mL
	$C = 1/H_j * z$	titer (the concentration of the volumetric solution is stored as auxiliary value H_j)
c.	<u>Volume and weight of sample unknown</u>	<u>Unit</u>
	$C = M/z$	mg
	$C = 1/z$	mmol

8.6.3 Nominal content

a. Dispensing / Predispensing

If you select the parameter "% nominal content" under the **Titration** function with titration modes DOS, EQP and EP to predispense or dispense you must specify three values:

- the amount to be dispensed in % of the absolute nominal consumption (metered amount)
- the nominal content
- the conversion constant: The conversion constant C converts mmol to the unit of the nominal content.

The volume that is predispensed or dispensed depending on the nominal content of a substance is calculated from the following formula:

$$\frac{\% \text{ of absolute nominal consumption}}{100\%} * \frac{\text{nominal content of sample}}{\text{conversion constant}} * \frac{\text{weight or volume}}{c * t}$$

Example 1: You determine the titer of a 0.1 mol/L NaOH solution with potassium hydrogen phthalate (M = 204.2 g/mol) and weigh in 0.1954 g.

The predispensing should be 80% of the absolute mL consumption. You enter the number **80**.

You expect a titer of 1. Enter the number **1**.

The conversion constant for the titer calculation is: **M/(1000 * z)**. Select this formula from the recommendation menu.

$$V = \frac{80\%}{100\%} * \frac{1}{0.2042 \text{ g/mol}} * \frac{0.1954 \text{ g}}{0.1 \text{ mol/L}}$$

7.665 mL are predispensed with these entries.

Example 2: You determine the % content of 0.085 g NaCl ($M = 58.44 \text{ g/mol}$) with 0.1 mol/L AgNO_3 (titer = 0.992).

The predispensing should be 85% of the absolute mL consumption. Enter the number **85**.

You expect a nominal content of 100%. Enter the number **100**.

The conversion constant for this content determination is: **$M/(10 * z)$** . Select this formula from the recommendation menu.

$$V = \frac{85\%}{100\%} * \frac{100\%}{5.844 \text{ g/mol}} * \frac{0.085 \text{ g}}{0.0992 \text{ mol/L}}$$

12.46 mL AgNO_3 are predispensed with these entries.

Example 3: You determine the content in g of 5 mL of a KOH solution ($M = 56.11 \text{ g/mol}$) with 0.1 mol/L HCl (titer = 0.981).

The predispensing should be 90% of the absolute mL consumption. Enter the number **90**.

You expect a nominal content of 5 g KOH/L. Enter the number **5**.

The conversion constant for this content determination is: **M/z** . Select this formula from the recommendation menu.

$$V = \frac{90\%}{100\%} * \frac{5 \text{ g/L}}{56.11 \text{ g/mol}} * \frac{5 \text{ mL}}{0.0981 \text{ mol/L}}$$

4.088 mL HCl are predispensed with these entries.

Example 4: You determine the active substance of a tablet with 1 mol/L NaOH (titer = 1.026). The amount of active substance is specified with 20 mmol/tablet. The sample weight is thus 1.

The predispensing should be 70% of the absolute mL consumption. Enter the number **70**.

You expect a nominal content of 20 mmol. Enter the number **20**.

The conversion constant for this content determination is **1**.

$$V = \frac{70\%}{100\%} * \frac{20 \text{ mmol}}{1} * \frac{1}{1.026 \text{ mol/L}}$$

13.65 mL NaOH are predispensed with these entries.

b. Termination after nominal consumption

If you select this parameter under the **Titration** function with titration mode EQP, you must define three values:

- the termination volume in % of the absolute nominal consumption; the added volume must be, e.g. 10% greater than the nominal consumption to the equivalence point
- the nominal content
- the conversion constant.

The volume that is used as a termination criterion depends on the nominal content of a substance and is calculated using the same formula as above.

Example: You titrate the weight percent of Cl^- ($M = 35.45 \text{ g/mol}$) of a salt solution (0.924 g) that contains various chlorides with 0.1 mol/L AgNO_3 (titer = 0.992).

You would like to abort the titration when you have titrated to 10% above the expected mL consumption, that is 110%. Enter the number **110**.

You expect a chloride fraction of 50 mg/g. Enter the number **50**.

The conversion constant for this content determination is **M/z**. Select this formula from the recommendation menu.

$$V = \frac{110\%}{100\%} * \frac{50 \text{ mg/g}}{35.45 \text{ g/mol}} * \frac{0.924 \text{ g}}{0.0992 \text{ mol/L}}$$

14.45* mL AgNO_3 are titrated with these entries before the titration is terminated.

* If the calculated amount falls between two increments, the next increment will still be added. Depending on the increment size, a volume of 14.47 mL, for example, can thus be titrated.

8.6.4 Formulae for limiting the equivalence point

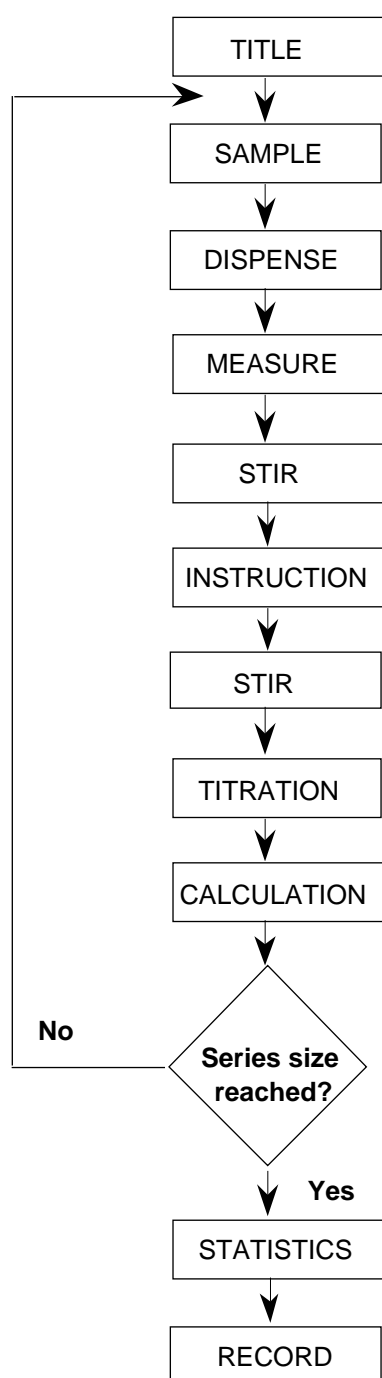
To calculate the consumption of titrant for a specific equivalence point in a titration curve with several equivalence points, you can have **Q** identified by a condition, such as:

$R1 = Q(EPOT > -50)$	Titrant consumption up to the 1st equivalence point of the 1st Titration function. Equivalence points with a potential under -50 mV (e.g. -51...-300) will not be considered.
$R1 = Q(EPOT > -50)[2]$	Titrant consumption up to the 1st equivalence point of the 2nd Titration function. Equivalence points with a potential under -50 mV (e.g. -51...-300) will not be considered.
$R1 = Q(EPOT \sim 100)[2]$	Titrant consumption up to the 1st equivalence point of the 2nd Titration function. The equivalence point with a potential value closest to 100 will be determined.
$R1 = Q(EPOT \sim P1)$	Titrant consumption up to the 1st equivalence point of the 1st Titration function. The equivalence point lying closest to the buffer potential P1 will be determined.
$R1 = Q(EPOT \sim H11)$	Titrant consumption up to the 1st equivalence point of the 1st Titration function. The equivalence point lying closest to the auxiliary value stored under H11 will be determined.
$R1 = Q(P1 < EPOT < 300)$	Titrant consumption up to the 1st equivalence point of the 1st Titration function. The equivalence point lying within the range of the buffer potential P1 and 300 mV will be determined.

8.7 Examples of methods

Note: Numerous applications performed with the DL70 titrator (predecessor to the DL70ES) are available in brochures (see Section 11.3.2).

8.7.1 Series titration with functions that the titrator performs before the actual sample addition



The **Sample** and **Statistics** functions are the identifiers for the loop of a series.

Sample: You specify all parameters. After starting the method you confirm the sample data mask with **RUN** without entering the weight (volume).

Dispense: The specified volume of a titrant should be added.

Measure: The titrator should acquire the measured value E in accordance with the specified conditions.

Stir: The titrator should not stir when you add the sample: The speed = 0%, the time = 0 so that it immediately starts with the next function.

Instruction: The entered request "Add sample" appears supported by an audio signal. You confirm the instruction with **RUN** after sample addition.

Stir: The titrator must stir and first dissolve the added sample: The speed = 70%, the time = 30 s.

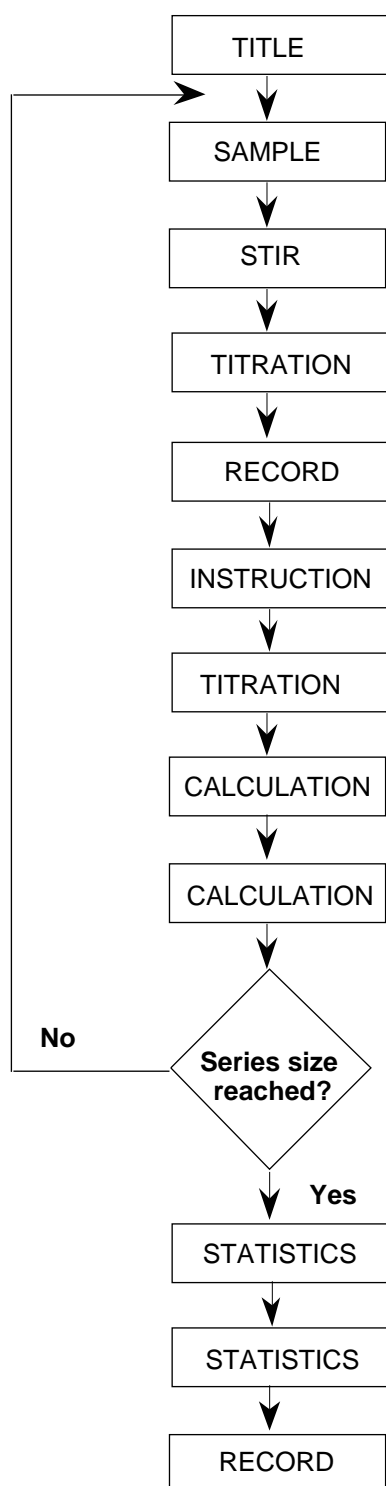
Titration: The titrator starts to execute the Titration function. If you have specified a predispensing to nominal content, the titrator first requests entry of the weight.

Calculation: If you have not yet entered the weight, the titrator requests the values when it executes this function.

Note: If you add a *Calculation* function with R = m after the Instruction function, the titrator requests the weight immediately.

8.7.2 Method with 2 **Titration** functions

Determination of the acetic acid and chloride contents in ketchup



Sample: $n = 3$, lower weight limit = 1.0 g, upper weight limit = 3.0 g, $M = 60.01$, $z = 1$. (Always enter the molar mass and the equivalent number of the substance that is determined first.)

Stir: Speed = 80%, time = 60 s: The titrator stirs for 60 s to ensure proper suspension of the ketchup before starting the **Titration** function.

Titration: Titrant = 0.1 mol/L NaOH, sensor = DG111-SC, unit of measurement = mV, titration mode EQP.

Record: If you wish to have a record of the titration curve and the table of measured values of this titration, you must add the **Record** function here. The titrator will not store these data if a **second Titration** function follows.

Instruction: The inputted request "Add H_2SO_4 " appears. The pH of the solution must be lowered for the chloride determination. Confirm the instruction with **RUN**.

Titration: Titrant = 0.1 mol/L $AgNO_3$, sensor = DM141-SC, unit of measurement = mV, titration mode EQP.

Calculation: $R = Q * C/m$, $C = M/(10 * z)$, unit = %.

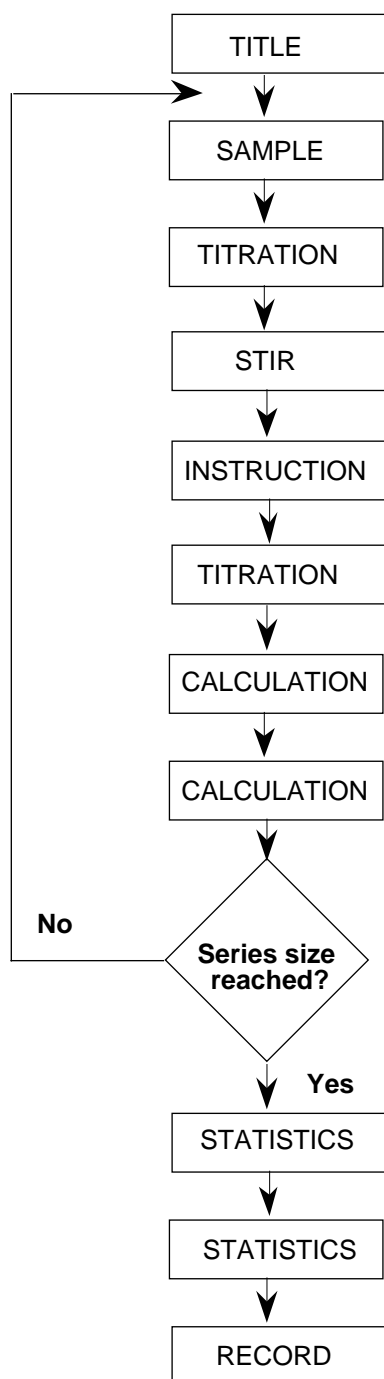
Calculation: $R2 = Q[2] * C2/m$, $C2 = 3.55 (M/(10 * z))$, unit = %. (The constant for the determination of chloride must now be entered as numerical value.)

Statistics: R_i (i = index) = R . The mean value of the first result (first **Calculation** function) of the three determinations of the acetic acid content is calculated.

Statistics: R_i (i = index) = $R2$. The mean value of the second result (second **Calculation** function) of the three determinations of the chloride content is calculated.

8.7.3 If your sample contains substances whose determination requires a new sample each time, compile a method that comprises **submethods**, in other words a method with **two** or **more Sample** functions.

Single determination of Ca^{2+} content, the sum of Ca^{2+} and Mg^{2+} in drinking water with the calculation of the American, French and German degree of hardness and the calculation of the Mg^{2+} content.



Sample: $n = 3$, fixed volume = 50 mL, $M = 40.08$, $z = 1$. The titrator begins to stir as soon as it has executed this function.

Titration: Titrant = 1.0 mol/L NaOH, sensor = DG111-SC, unit of measurement = pH, titration mode DOS. Titration to pH = 12.

Stir: Speed = 80%, time = 120 s. The titrator should stir for 2 min to ensure complete precipitation of the $\text{Mg}(\text{OH})_2$.

Instruction: The inputted request "Add indicator solution" appears. Confirm the instruction with **RUN**.

Titration: Titrant = 0.1 mol/L EDTA, sensor = DP660/550, unit of measurement = mV, titration mode EQP.

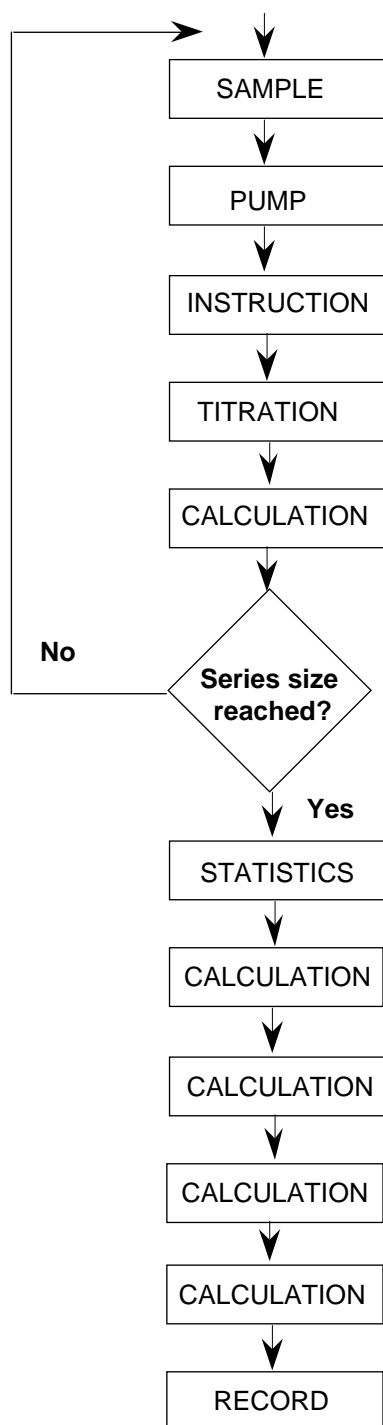
Calculation: $R = Q[2] * C/U$, $C = M * 1000/z$, unit = mg/L.

Calculation: $R2 = Q[2]$ (titrant consumption in mmol of the second **Titration** function). Delete the constant with **CE**.

Statistics: R_i (i = index) = R . The mean value of the first result (first **Calculation** function) of the three Ca^{2+} determinations is calculated.

Statistics: R_i (i = index) = $R2$. The mean value of the second result (second **Calculation** function) for the mmol consumption is calculated.

Record: If you wish to have a record of the titration curve and the table of measured values for this titration you must add the function here.



Sample: $n = 3$, fixed volume = 50 mL. The titrator starts to stir at the default speed as soon as it has executed the function.

Pump: Auxiliary reagent = NH_3 buffer (pH 10), volume = 5 mL.

Instruction: The inputted request "Add indicator solution" appears. Confirm the instruction with **RUN**.

Titration: Titrant = 0.1 mol/L EDTA, sensor = DP660/550, unit of measurement = mV, titration mode EQP.

Calculation: $R3 = Q[3]$ (titrant consumption in mmol of the third **Titration** function). Delete the constant with **CE**.

Statistics: R_i (i = index) = $R3$. The mean value of the third result (third **Calculation** function) for the mmol consumption is calculated.

Calculation: $R4 = \bar{x}[3] * C2/U$,
 $C2 = 100.09 * 1000/1$ ($M_{\text{CaCO}_3} * 1000/z$ for the American degree of hardness: ppm CaCO_3).

Calculation: $R5 = \bar{x}[3] * C3/U$,
 $C3 = 100.09 * 100/1$ ($M_{\text{CaCO}_3} * 100/z$ for the French degree of hardness: 10 mg CaCO_3/L).

Calculation: $R6 = \bar{x}[3] * C4/U$, $C4 = 56.08 * 100/1$
 $(M_{\text{CaO}} * 100/z$ for the German degree of hardness: 10 mg CaO/L).

Calculation: $R7 = (\bar{x}[3] - \bar{x}[2]) * C5/U$ gives the Mg^{2+} -content, $C5 = 24.31 * 1000/z$;
 $\bar{x}[3]$ is the mean value of the 3rd **Statistics** function
 $\bar{x}[2]$ is the mean value of the 2nd **Statistics** function.

Note: You must enter numerical values for the constants of these 4 calculation functions.

8.8 **Scheme for method design**

You can copy this scheme to set up functions with their parameters for a new method before you enter it in the Editor menu.

<div>TITLE</div>	Method ID: Title:	
<div>SAMPLE</div>	Number samples: Titration stand: Entry type: ID1:	Molar mass M: Equivalent number z: Temperature sensor:
<div></div>		
<div></div>		
<div></div>		
<div></div>		
<div></div>		
<div></div>		
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Contents	Page
9. Error messages and malfunctions	9-3
9.1 Error messages of the titrator	9-3
9.2 Other errors and malfunctions	9-5

9. Error messages and malfunctions

9.1 Error messages of the titrator

The titrator sends you messages regarding errors that you can rectify yourself. Four such messages draw your attention to this section:

1. EEPROM inserted wrongly (EEPROM = user data memory)

For some reason or other, you have inserted the user data memory in which the installation data are stored into the second IC socket (see Section 11.1.8: *Illustration IC socket (2)*).

- Measure:**
- Switch off the titrator and disconnect from power supply.
 - Insert the user data memory into the first IC socket.

2. Faulty data deleted

- a. The titrator has saved only parts of a method if, e.g. during storage of this method the power failed. It deletes this method completely. It is also possible that several methods have been deleted.

- Measure:**
- Confirm the message with **RUN**.
 - Check whether your methods have been deleted and reenter if necessary.

- b. The titrator has saved only parts of parameters of a resource if, e.g. during storage of these parameters the power failed. It deletes the entire list of this resource (e.g. all titrants or all sensors).

- Measure:**
- Confirm the message with **RUN**.
The titrator now loads the standard list of the resource, e.g. all titrants stored in the titrator on delivery.
 - Check what list has been changed:
If the user data memory is too small, the titrator stores only the titrants for which it has space.
If the memory is full, the titrator does not store any titrant. In this case, you must delete other installation data or one of your methods to create memory space.
 - Then switch the titrator off and on again.
 - Check that all resources are again present.

If you are frequently shown the error message, you should contact METTLER service.

3. Storage not possible

- a. The titrator can **not** assign the titer, auxiliary value or the calibration parameters to the appropriate resources as their list is missing (titrant, sensors or auxiliary values).

Measure: – Confirm the message with **RUN**.

- Check whether the list of the resource has been deleted.
- Switch the titrator off then on again: The titrator reloads the standard list of the corresponding resource, e.g. all the titrants stored in the titrator on delivery.

If the user data memory is too small, the titrator stores only the titrants for which it has space.

If the memory is full, the titrator does not store any titrant. In this case, you must delete other installation data or one of your methods to create memory space.

- Then switch the titrator off and on again.
- Check that all resources are again present.

- b. The titrator cannot, for example, save data if the user data memory is full. This is possible

- for auxiliary reagents you want to add in INSTALLATION,
- for a method you create in EDITOR,
- for sample data you want to enter in the method list for methods in ANALYSIS,
- for "permanently" saving a modification of a currently running method,
- for "permanently" saving the evaluation criteria of the Titration function of a currently running method (parameter: "Stop for reevaluation").

Measure: – Confirm the message with **RUN**.

- Delete method or installation data, or insert a second user data memory.

- c. The titrator is controlled by a computer and is performing a **learn method**. The EQP or EP parameters of the Titration function are **not saved** by the titrator.

Measure: – Confirm the message with **RUN**.

- Always perform a learn method with a method stored in the titrator!

If you are frequently shown the error message, you should contact METTLER service.

4. Memory faulty

Parts of the user data memory are faulty.



Measure: – Call METTLER service to have the memory changed.

In the meantime, you can continue working with the titrator.

9.2 Other errors and malfunctions

The following is a listing of faults and malfunctions which are not reported by the titrator and should help you rectify many of the possible malfunctions yourself thereby reducing your dependency on the METTLER service.

Note: Before you call METTLER service, please print out the system information, which provides details of the equipment configuration of the titrator, and inform the service of these:

press  +  : The information will be printed out.

Fault	Possible cause	Corrective measure
No display on titrator	Instrument not connected to power Fuse defective	Plug in instrument Check fuse and replace if necessary
A few dots of the display are missing		Call METTLER service
The display does not match the key pressed		Call METTLER service
Stirrer will not stir	Sensors can block stirrer at the titration stand	Check placement of sensors
Units attached to the outputs are inoperative	Auxiliary instrument defective	Check instrument at another auxiliary output Call METTLER service
Transfer error to attached peripheral	Peripheral faulty	Check attached units for proper functioning
Unit (printer, balance, terminal) at serial interface does not respond	Unit not switched on Wrong installation data Wrong configuration (switch settings)	Switch on unit Installation data and configuration must match (see Section 1.8)

Fault	Possible cause	Corrective measure
Burette does not move to zero position when instrument switched on	Burette drive faulty	Check burette drive at another station Call METTLER service
Wrong potential or pH values	Electrode defective	Check electrode (see electrode sheet) Check installation data Use new electrode
No dispensing, titrant discharged from stopcock or piston	Burette tip blocked Follower cam at burette stopcock installed wrongly	Clean burette tip Insert follower cam correctly (see Section 11.1.2.3)

Contents	Page
10. Applications	10-3
10.1 List of the METTLER methods	10-4
10.2 Water determination according to Karl Fischer	10-5
10.2.1 Safety measures	10-5
10.2.2 Startup	10-5
10.2.3 Description of the four Karl Fischer methods	10-7
10.2.3.1 MFK1 KF Titration	10-7
10.2.3.2 MFK2 KF Drift Determination	10-7
10.2.3.3 MFK3 KF Standby Titration	10-8
10.2.3.4 MFK4 KF Titer with Na-Tartrate	10-8
10.2.4 Limits of detection and reproducibility	10-9
10.2.5 Performing Karl Fischer titrations	10-9
10.2.5.1 Procedure A: Pretitration, standby titration and drift determination	10-9
10.2.5.2 Procedure B: Pretitration, drift and titer determination	10-10
10.2.5.3 Procedure C: Pretitration, drift determination and titration of a single sample	10-10
10.2.5.4 Titration of series	10-11
10.2.6 Adapting function parameters	10-12
10.2.7 Drift compensation	10-12
10.2.8 Possible problems, their cause and rectification	10-13
10.2.9 A few examples of results	10-14
Application sheets	M001 - M016 MKF1 - MKF4

10. Applications

In this section you will find application sheets for all methods that we have developed and stored in the titrator as METTLER methods. The four methods you need to determine moisture by the Karl Fischer method are described in greater detail.

Methods M002 - M016 include the calibration of pH electrodes and titer determinations which you should always perform before you analyze your samples with the appropriate titrant. At the same time, these methods serve as a model for the development of your own methods:

- You can examine the parameters of the individual functions.
- You can adopt control parameters of the **Titration** function such as titrant addition and measure mode for the same titration reaction by either modifying the METTLER method and storing it as a user method or developing a new method with the aid of the standard method (see Sections 2.1 and 2.1.3).

10.1 List of the METTLER methods

M001: Acid Content

M002: Titer of NaOH (0.1 mol/L)

M003: Titer of HCl (0.1 mol/L)

M004: Calibration pH Electrode

M005: Titer of HClO₄ (0.1 mol/L)

M006: Titer of AgNO₃ (0.1 mol/L)

M007: Titer of EDTA (0.1 mol/L)

M008: Titer of Fe(II) (0.1 mol/L)

M009: Titer Na₂S₂O₃ (0.1 mol/L)

M010: Titer of TBAH (0.1 mol/L)

M011: Titer $\frac{1}{2}$ H₂SO₄ (0.1 mol/L)

M012: Titer of CPC (0.01 mol/L)

M013: Titer of KMnO₄ (0.1 mol/L)

M014: Titer of EGTA (0.1 mol/L)

M015: Titer Ce(SO₄)₂ (0.1 mol/L)

M016: Titer of $\frac{1}{2}$ I₂ (0.1 mol/L)

MKF1: KF Titration

MKF2: KF Drift Determination

MKF3: KF Standby Titration

MKF4: KF Titer with Na-Tartrate

10.2 Water determination according to Karl Fischer

You can use the titrator for the reliable determination of the water content of substances when it is greater than 2 mg H₂O per sample.

10.2.1 Safety measures

All Karl Fischer reagents are readily inflammable solutions. They are toxic:

- Do not inhale and avoid skin contact!
- On skin contact, immediately wash off with copious amounts of water!
- On eye contact, immediately irrigate with copious amounts of water, then consult a physician!

10.2.2 Startup

You will find general explanations regarding burettes and titration stand in Section 11.1. This section deals only with specific points.

Accessories

The Karl Fischer accessories are listed in Section 11.3, page 11-36.

Burette

In METTLER methods MKF1 - MKF4, a **5 mL** burette is installed on burette drive **3** for the Karl Fischer titrant.

- To protect the titrant against moisture, mount the drying tube holder in the opening of the burette holder,
- fill the drying tube with molecular sieves or some other drying agent and position on the drying tube holder (see *Accessories*, page 11-30).

Caution: KF titrants evolve gas (SO₂) especially at elevated temperatures, causing bubbles to form in the tubing as well as in the burette (stopcock). For this reason you should rinse burettes prior to titration!

Titration head

- Always insert the electrode and burette tip diagonal to each other so that the solution is thoroughly mixed before its potential is measured by the electrode.
- To dispense the KF solvent, take the appropriate measures (see Section 10.2.3.4: Note a.).

- Insert the gas inlet if you wish to titrate under a dry inert gas.
- Insert the septum stopper if you wish to add liquids using a syringe.
- Close the remaining openings with stoppers.

The weighing spoon is used to add solids.

The drain cock serves to empty the titration vessel.

Double-pin platinum electrode

- Attach the electrode to the polarization current source DK102A.
- Attach a triaxial cable to the current source and plug it into sensor input 1 (sensor input 2 is less suitable).

Polarization current source

- Switch on the current source only when the platinum tips of the electrode are completely covered by solvent.
- Never set the current higher than 1 μA . Normally, 0.2 – 0.5 μA suffice.

It is important to have an initial potential of at least -170 mV! (If not, set a higher current.)

- Always switch off the current source when not in use!

Karl Fischer reagents

With METTLER methods MKF1 - MKF4, we have worked with the pyridine-free KF reagents from Riedel-de Haen (HYDRANAL[®]), Merck, Fluka and J.T. Baker (ReAquant[®]) and used both one- and two-component reagents.

Solvents

With the one-component reagent, methanol is used as the solvent. To improve the solubility of samples, we also used solvent mixtures of methanol with chloroform, formamide, toluene or 1-decanol. In these mixtures the proportion of methanol should be greater than 50% (exception: with chloroform, greater than 20%).

In the case of the two-component reagent, the KF solvent is used as solvent. Again, we have added other solvents such as methanol, chloroform, formamide, toluene and 1-decanol. The proportion of KF solvent should be above 50% in these mixtures.

10.2.3 Description of the four Karl Fischer methods

Four methods are stored in the titrator for the KF titration:

10.2.3.1 MKF1 KF Titration

For the titration of the solvent (pretitration) or for the titration of a sample

This method has two **Titration** functions:

First Titration function

Rough titration for the rapid titration of 80 - 90% of the water content of a solvent or sample.

- Titration to absolute end point with dynamic titrant addition (see Section 2.3.12.3). The absolute end point (EPA) is -5.0 mV.
- The wait time between the increments is 2 - 4 s, the increment size 0.08 - 0.2 mL.

Second Titration function

Fine titration for titration of the water content of the solvent or sample.

- Titration to absolute end point with dynamic titrant addition. The absolute end point (EPA) is -5.0 mV.
- The wait time between the increments is 7 - 7.5 s, the increment size 0.015 - 0.02 mL.

We have selected the parameters for both Titration functions so that the use of different solvents has no appreciable influence on the titration progress.

No titrant consumption in the **second** Titration function (fine titration) means **overtitration** in the **first** Titration function. The display then shows you how to proceed with the message *Overtitrated!*: You must set the EPA of the first Titration function lower!

10.2.3.2 MKF2 KF Drift Determination

With solution titrated to dryness, a small titrant consumption can always be detected, and this is due to moisture diffusing into the system. You must measure and store this drift in order to take it into account in the content calculation of a sample.

The MKF2 method contains two **Titration** functions, separated by a wait time of ten minutes. The **first** Titration function is used to titrate the solution to dryness, the **second** Titration function to determine the drift, which is then stored under auxiliary value **H20**.

Note: a. The drift value depends on the tightness of the titration stand and the atmospheric moisture and can be between 10 and 100 $\mu\text{g}/\text{min}$.

Note: b. We have selected a drift determination time of 10 minutes as a compromise between speed and accuracy: With 10 minutes the relative standard deviation of the drift is 5%, with 5 minutes 12%.

Caution: Do not select this method to titrate water in fresh solvent!

10.2.3.3 MKF3 KF Standby Titration

For the continuous titration of ingressing moisture

The method contains an **mV-stat** function that maintains the potential at -20 mV. This potential is an empirical value which, irrespective of titrants, solvents and samples, keeps the titration vessel dry.

The stating is limited (55 h). To perform a titration or drift determination, you must always terminate the standby titration with **RESET**.

Caution: Do not select this method to titrate water in fresh solvent!

10.2.3.4 MKF4 KF Titer with Na-Tartrate

Titer determination of the KF titrant with sodium tartrate • 2 H₂O

The method contains the same two **Titration** functions as method MKF1 and also the **Titer** function, which is used to store the determined value in the parameter mask of the titrant.

- Notes: a. Dispensing of the solvent is not defined in the MKF1 and MKF2 methods. If your titrator has two (several) burette drives, we advise
- installing the KF solvent for a 20 (10) mL burette,
 - adding the **Dispense** function to MKF1 or MKF2 and storing this as a user method.

You thus above all avoid ingress of moisture into the titration vessel through solvent addition.

- b. As a calibration substance you can also use deionized H₂O or a standard water solution of 5 mg H₂O/mL (modify parameters of the *Sample* function and the fifth *Calculation* function accordingly!):

H₂O: by means of a microliter syringe approx. 10 µL, accurate to at least 0.5%.

Standard water solution: approx. 2 mL, accurate to at least 0.5%.

10.2.4 Limits of detection and reproducibility

Limit of detection: 0.6 mg water (for titrant "5 mg H₂O/mL").

Reproducibility: <1% relative standard deviation (RSD) for samples with a water content of 15 - 25 mg/sample.

To achieve this reproducibility, the sample must contain at least 15 mg water.

10.2.5 Performing Karl Fischer titrations

10.2.5.1 Procedure A: Pretitration, standby titration and drift determination

- Add 40 mL methanol or KF solvent to the titrant vessel (see Note a. in Section 10.2.3.4).
- Start method **MKF1** to titrate the solvent to dryness (so-called **pretitration**).
- Enter **1** for sample weight and number of samples.
- On completion of the pretitration, start KF standby titration **MKF3** to condition the titration vessel: A newly installed titration vessel is still not completely dry after the pretitration.

Note: If the titration vessel is already conditioned, you can immediately perform the drift determination **MKF2**.

- Allow method **MKF3** to run until the drift is stable. This normally takes 2 hours. We recommend letting the standby titration run overnight.

Note: For the standby titration to follow the pretitration automatically,

- enter methods MFK1 and MFK3 in the method list,
- select titration stand "Auto stand" instead of "Stand 2" (see Section 1.7) and
- start **List once**.

(With the DL67 this is not possible.)

- Terminate the standby titration with **RESET**.
- Start the drift determination **MKF2**.

Note: You should perform drift determinations fairly often to check the value.

10.2.5.2 Procedure B: Pretitration, drift and titer determination

- After the first drift determination, empty the titration vessel, add 40 mL methanol or KF solvent (see Note a. in Section 10.2.3.4) and start method **MKF1** to titrate the solvent to dryness.
- Start method **MKF2** to check the drift.
- Start method **MKF4** to determine the titer of the KF titrant.

Notes: a. The concentration of the KF titrant has been set at 5 mol/L, corresponding to 5 g H₂O/L. (The titrator does not recognize the unit g/L for titrants.)

b. To achieve good reproducibility, the initial weight of sodium tartrate should be 0.09 - 0.12 g ±0.1 mg (see also Section 10.2.4).

c. In the titer determination, you must take into account the limited solubility of sodium tartrate in methanol and chloroform:

In 40 mL methanol you can perform max. 4 titer determinations.

In 1:1 mixtures of chloroform/methanol the titer determination with sodium tartrate is not possible since it does not completely dissolve.

The solubility of sodium tartrate is very good in the KF solvent of the two-component reagent.

d. If methods MKF1, MKF2 and MKF3

- are entered in the method list,
- "Auto stand" is defined as the titration stand (see Section 1.7) and
- **List once** is started,

the titrator executes the pretitration followed by the drift determination. Afterwards it will start the standby titration.

- To determine the titer after this, terminate the standby titration with **RESET**. (For the DL67 this is not possible.)

10.2.5.3 Procedure C: Pretitration, drift determination and titration of a single sample

- Add 40 mL methanol or KF solvent (see Note a. in Section 10.2.3.4) and start method **MKF1** to titrate the solvent to dryness.
- Start method **MKF2** to check the drift.
- Then **immediately** start method **MKF1** to determine the water content of the sample.

Notes: a. If you interrupt the individual determinations, you should start the **MKF3** standby titration to ensure that the titrant vessel remains free from water.

b. To achieve good reproducibility, the sample should contain 15 - 25 mg water (see Section 10.2.4).

Notes: c. The stirring time and speed of the Stir function in method MKF1 are 10 s and 50%, respectively. If a longer stirring time or a higher speed are necessary, you must change both parameters and save the method as a user method.

d. If methods MKF1, MKF2 and MKF3

- are entered in the method list,
- "Auto stand" is defined as the titration stand (see Section 1.7) and
- **List once** is started,

the titrator executes the pretitration followed by the drift determination. Afterwards it will start the standby titration.

– To titrate a sample after this, terminate the standby titration with **RESET**.

(For the DL67 this is not possible.)

10.2.5.4 Titration of series

The MKF1 method also allows a sample series to be run:

- After the start of the MKF1 method, enter the number of samples.
- Perform the individual titrations of the series in succession.

Notes: a. The time interval between the individual titrations must be as small as possible since some moisture can always diffuse into the titration vessel during this time and the drift can be compensated only by calculation.

b. You can not change the solvent within a series. The number of samples of a series depends on the capacity of the solvent: With unproblematic samples (no interference by the matrix), it is approx. 100 mg H₂O/20 mL solvent, depending on the solvent used. Attainment of the capacity limit is shown by a slowdown in the reaction rate.

10.2.6 Adapting function parameters

MKF2 and MKF3

In the MKF2 drift determination and MKF3 standby titration, you do not need to modify any parameter values. These methods are optimum for all pyridine-free titrants and solvents.

MKF1 and MKF4

If the titrator overtitrates in your determinations or the complete titration takes too long, all you have to do is adapt the absolute end point of the **first** Titration function.

Karl Fischer reagent	EPA range of the first Titration function	Recommended EPA value
low reaction rate	–5 to –2 mV	–3 mV
moderate reaction rate, e.g. one-component reagent	–10 to –5 mV	–5 mV
high reaction rate, e.g. two-component reagent	–25 to –15 mV	–20 mV

In the optimum case, approx. 0.08 mL titrant are consumed in the **second** Titration function (fine titration). An acceptable range is 0.04 - 0.2 mL.

If the value lies outside this range, you can set the EPA of the **first** Titration function higher or lower **step by step**. The step size for the one-component reagent is 2 mV, for the two-component reagent 5 mV.

- Reagent consumption of the **second** Titration function **below 0.04 mL**:
Danger of overtitration → Step by step, set EPA of the first Titration function lower.
- Reagent consumption of the **second** Titration function **above 0.2 mL**:
Titration time >4 min → Step by step, set EPA of the first Titration function higher.

10.2.7 Drift compensation

The titrator acquires the running time for each processed method as the raw result TIME (see Section 3.1.3: Note a.). TIME is saved as auxiliary value H19 "before starting" (see method) and as H18 at the end of each sample determination. The difference between these values gives the titration time (see **3rd Calculation** function) and is used in the drift compensation (see **4th Calculation** function).

10.2.8 Possible problems, their cause and rectification

Problems	Possible cause	Measures
The titration does not start	DK102A not switched on	Switch on DK102A
Initial potential too high (above -170 mV)	Battery voltage of the DK102A current source too low Contaminated electrode Bent platinum tips	Check voltage, if need be change battery Clean with chromic acid and dry thoroughly
Drift value too high	Titration vessel not closed Titration vessel not completely dry	Check Allow standby titration to continue for a further 1-2 h, if need be overnight
Overtitrated	Contaminated electrode Poor mixing End point of the first Titration function too high	Clean with chromic acid and dry thoroughly Increase stirring speed Set end point 2 (5) mV lower (see Section 10.2.6).
Long titration time (sluggish approach to end point)	End point of the first Titration function too low Slow water loss from the sample Reaction of the sample with KF reagent (side reaction)	Set end point 2 (5) mV higher (see Section 10.2.6) Pulverize sample or perform external extraction Direct titration not possible
Infinite pretitration	Method MKF2 or MKF3 selected for the pretitration	Select method MKF1

Problems	Possible cause	Measures
Poor reproducibility	Initial weight too low Drift changes drastically Long wait times between the titrations without standby titration	Sample should contain at least 15 mg water Perform drift determination more frequently
First value of a series always too low or too high	Series after a standby titration	Start a drift determination before a series*

* **Explanation:**

The MKF1 and MKF3 methods do not have the same Titration functions (KF titration: **dynamic EP**; standby titration: **mV-stat**). The end point level is thus not identical. As a consequence, after a standby titration the first value of a series can differ.

10.2.9 A few examples of results

Titration with one-component reagent (EPA of the first Titration function: –5 mV),
Drift: 38 µg/min

Titer determination with sodium tartrate:	n = 2	\bar{x} = 1.07233	RSD = 0.4%
Titration: 10 µL water	n = 4	\bar{x} = 9.97 mg	RSD = 0.4%
Titration: 0.03 g cognac	n = 6	\bar{x} = 61.15 %	RSD = 0.5%

Titration with two-component reagent (EPA of the first Titration function: –20 mV),
Drift: 40 µg/min

Titer determination with sodium tartrate:	n = 2	\bar{x} = 1.0704	RSD = 0.9%
Titration: 10 µL water	n = 5	\bar{x} = 9.94 mg	RSD = 0.4%

11.1	Installation instructions for the titrator	11-3
11.1.1	Inserting the burette drive	11-3
11.1.2	DV1001, DV1005, DV1010, DV1020 burettes	11-5
11.1.2.1	Equipping the burette	11-6
11.1.2.2	Inserting the burette	11-7
11.1.2.3	Maintaining the burette parts	11-8
11.1.2.4	General notes	11-9
11.1.3	Equipping the titration stand	11-10
11.1.4	Rear view of the titrator	11-12
11.1.5	Power supply voltage, power fuse	11-14
11.1.6	Inserting an RS option	11-15
11.1.7	Setting and inserting a temperature option	11-16
11.1.8	Inserting a user data memory (IC)	11-18
11.2	Technical data	11-19
11.2.1	Measurement system	11-19
11.2.2	Auxiliary outputs	11-20
11.2.3	Burette drive modul	11-20
11.2.4	Interchangeable burettes	11-20
11.2.5	Propeller stirrer	11-20
11.2.6	Keypad	11-21
11.2.7	Display	11-21
11.2.8	Titration modes	11-21
11.2.9	Method concept	11-22
11.2.10	Memory	11-22
11.2.11	Attachment possibilities for peripherals	11-23
11.2.12	Additional data	11-24
11.3	Accessories	11-25
11.3.1	Standard equipment	11-27
11.3.2	Optional accessories	11-28

11.1 Installation instructions for the titrator

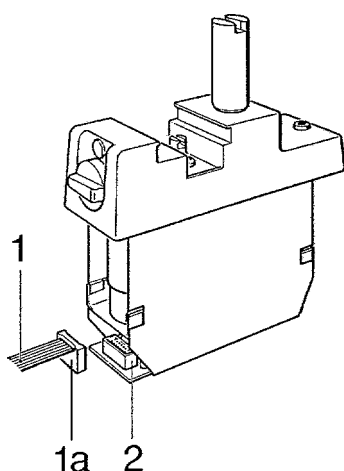
The aim of the following descriptions and drawings is to help you assemble the individual parts of the titrator and become acquainted with all interfaces and inputs and outputs for electrodes and stirrer.



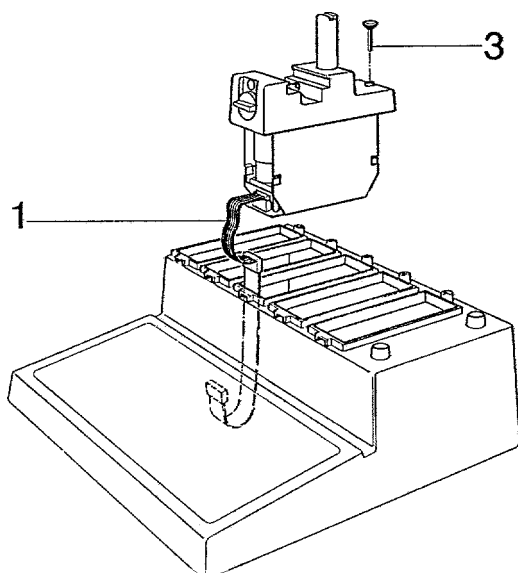
Risk of
electric shock

The titrator must be disconnected from all voltage sources before you remove the baseplate!

11.1.1 Inserting the burette drive



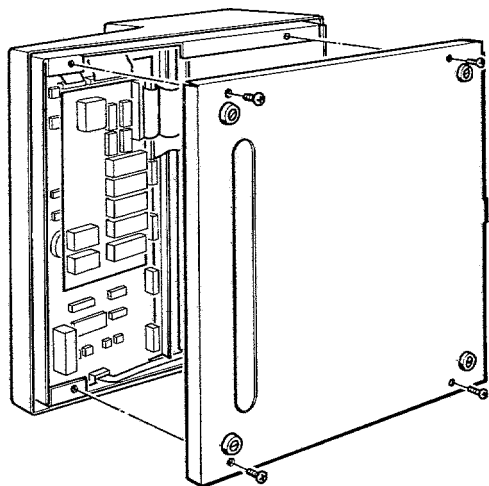
Plug connection (1a) of ribbon cable (1) – both connections are identical – into connector (2) of the burette drive.



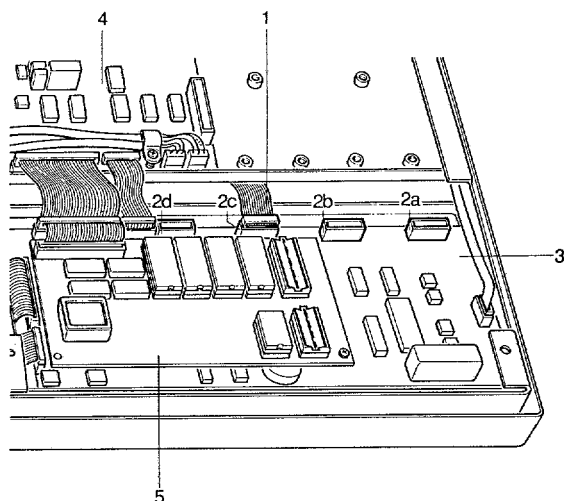
Place the burette drive on the third opening of the instrument – press ribbon cable (1) downward slightly to allow it to be led through the lower housing slot later – and fasten with screw (3).

Note

Please place the burette drive on the 3rd opening: For the first titration, which you perform according to the instructions given in the tutorial, the 3rd burette drive is defined in the method.



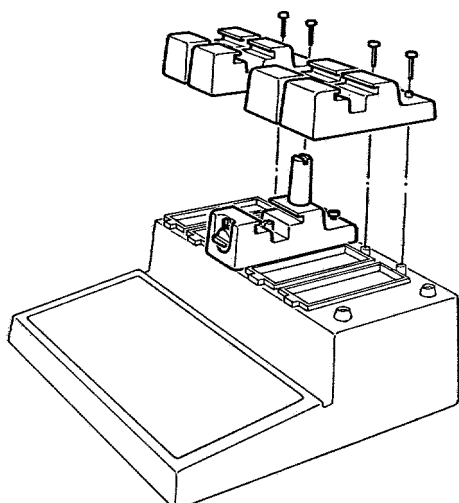
Lay the titrator on its side and undo the 4 screws to remove the baseplate.



Plug connection (1) of the ribbon cable of the burette drive into the third connector (2c) of the digital board (3).

(4) is the analog board, (5) is the processor board.

When you install additional burette drives, the first connector (2a) is allocated to burette drive 1, the second connector (2b) to burette drive 2, etc.



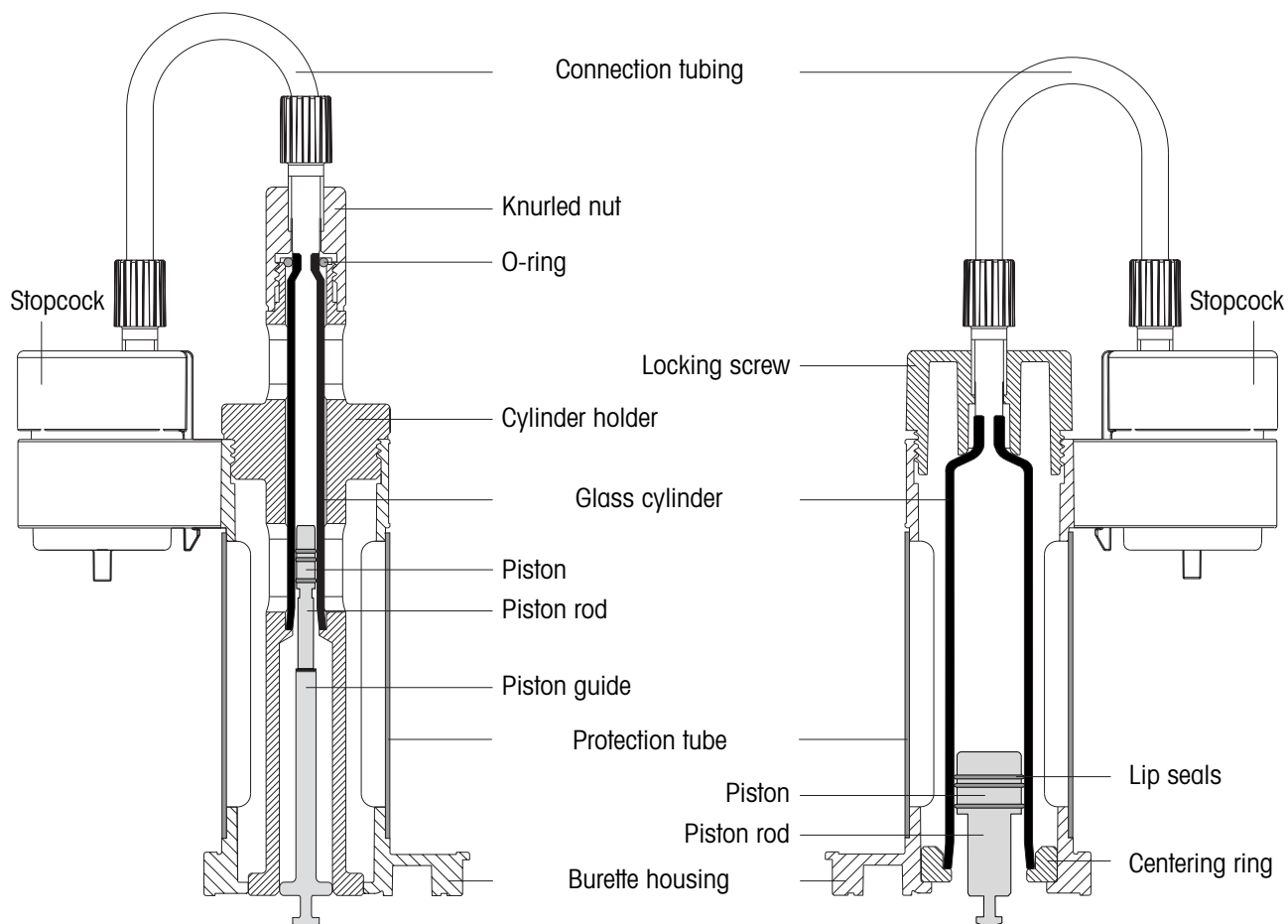
Screw on the baseplate and place the burette guides on the free openings.

11.1.2 DV1001, DV1005, DV1010, DV1020 burettes

The 5, 10 and 20 mL burettes differ only in the size of their cylinder, centering ring and piston, whereas the 1 mL burette has a different construction: its piston is longer, piston guide and cylinder holder "replace" the centering ring of the other burettes. Instead of the locking screw, its glass cylinder is fastened with an O-ring and a knurled nut.

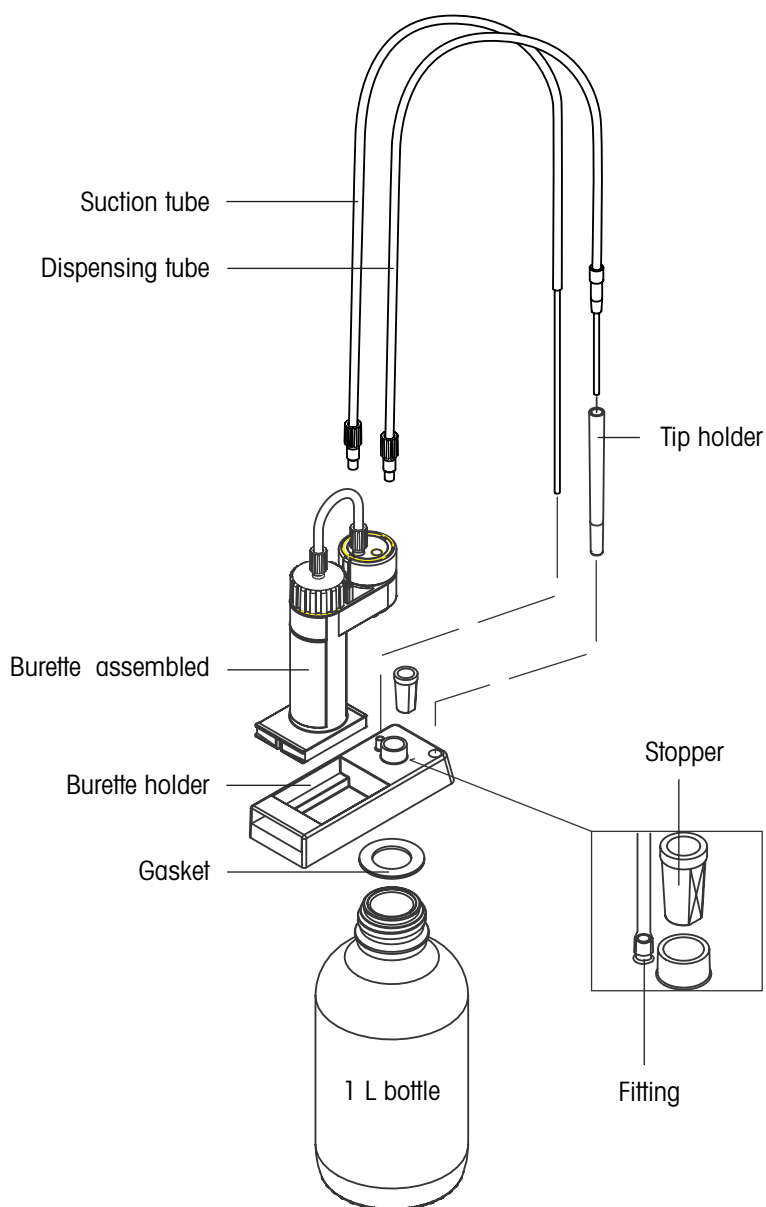
1 mL burette

5 / 10 / 20 mL burette

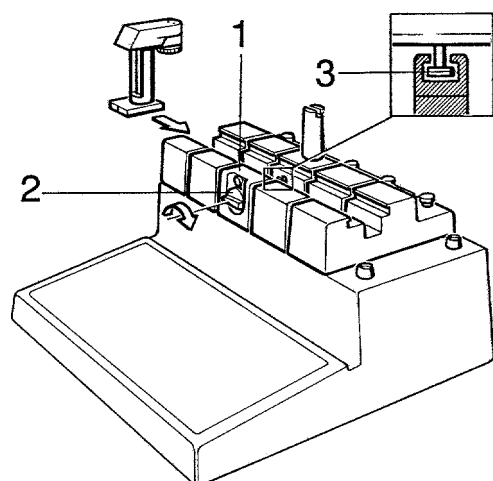


11.1.2.1 Equipping the burette

- Lay gasket on bottle and screw on burette holder.
- Insert stopper (or a drying tube with holder).
Caution: If you use a stopper as protection for the titrant, always use the stopper with flat side! Otherwise a partial vacuum forms in the bottle (order no. 23646).
- Push suction tubing into the bottle - slide the red PVC tubing over the fitting as kink protection - and fasten other end to left connection of burette head.
- Screw dispensing tubing into connection at right and place burette tip in tip holder.



11.1.2.2 Inserting the burette

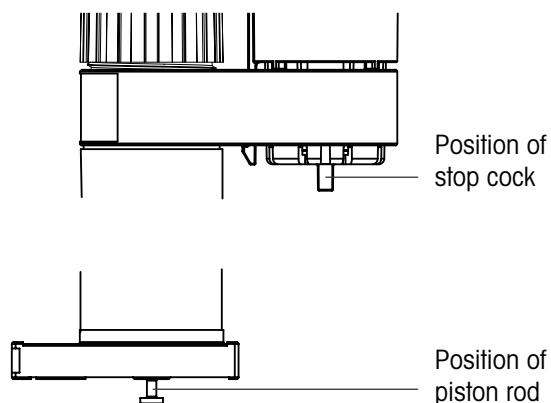


- Slide the burette onto burette drive 3 (1) with the stop of arrestment knob (2) located on left side.
- Check that piston rod is correctly positioned in push rod (3).
- Fix burette by turning the arrestment knob to the right.

Note

Before sliding the burette onto the titrator, check the exact position of the stopcock and the piston rod.

If the piston has been pushed too far into the cylinder, carefully take it out a short way. Then press the burette onto the burette holder thus positioning the piston exactly. Piston must project 7 mm!



11.1.2.3 Maintaining the burette parts

Depending on the titrant, you should clean the burette cylinder, piston, stopcock and tubing relatively often.

- Slide the burette off the titrator, invert it so that the stopcock points towards you and carefully take out the piston; this causes the burette contents to flow out through the suction tubing (waste or titrant bottle!).
- In the same position, turn the stopcock through 90° clockwise and any liquid in the stopcock will flow out through the dispensing tubing (waste bottle!).
- Unscrew the suction, dispensing and connection tubing.

1 mL burette

- Unscrew the knurled screw of the burette and remove the O-ring from the glass cylinder using tweezers.
- Unscrew the holder of the glass cylinder and take out the cylinder.

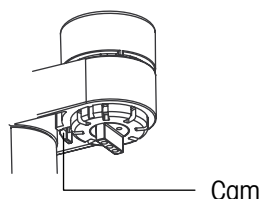
Caution: Do not misplace O-ring!

5, 10, or 20 mL burette

- Unscrew the locking screw of the burette and take out the glass cylinder.

Caution: Do not misplace the centering ring of the burette housing!

- Press the cam of the stopcock inward and lift the stopcock up and out.



- Depending on the contamination caused by the titrant, rinse cylinder and tubing with acids or deionized H₂O then with ethanol and finally dry the parts with oil-free compressed air or vacuum.
- Rinse the stopcock with solvents or deionized H₂O only! Then dry it with oil-free compressed air.
- Never place O-rings in organic solvents!
- Never attempt to remove any crystals in the cylinder by scratching with a hard object! Pipe cleaners or Q tips™ are more suitable.
- Never put the parts in a drying oven whose temperature is above 40 °C!
- Replace the piston if it leaks or is badly scored at the edge. Pay special attention to crystal formation between the lip seals of the piston if you work with NaOH/KOH and KF solutions!

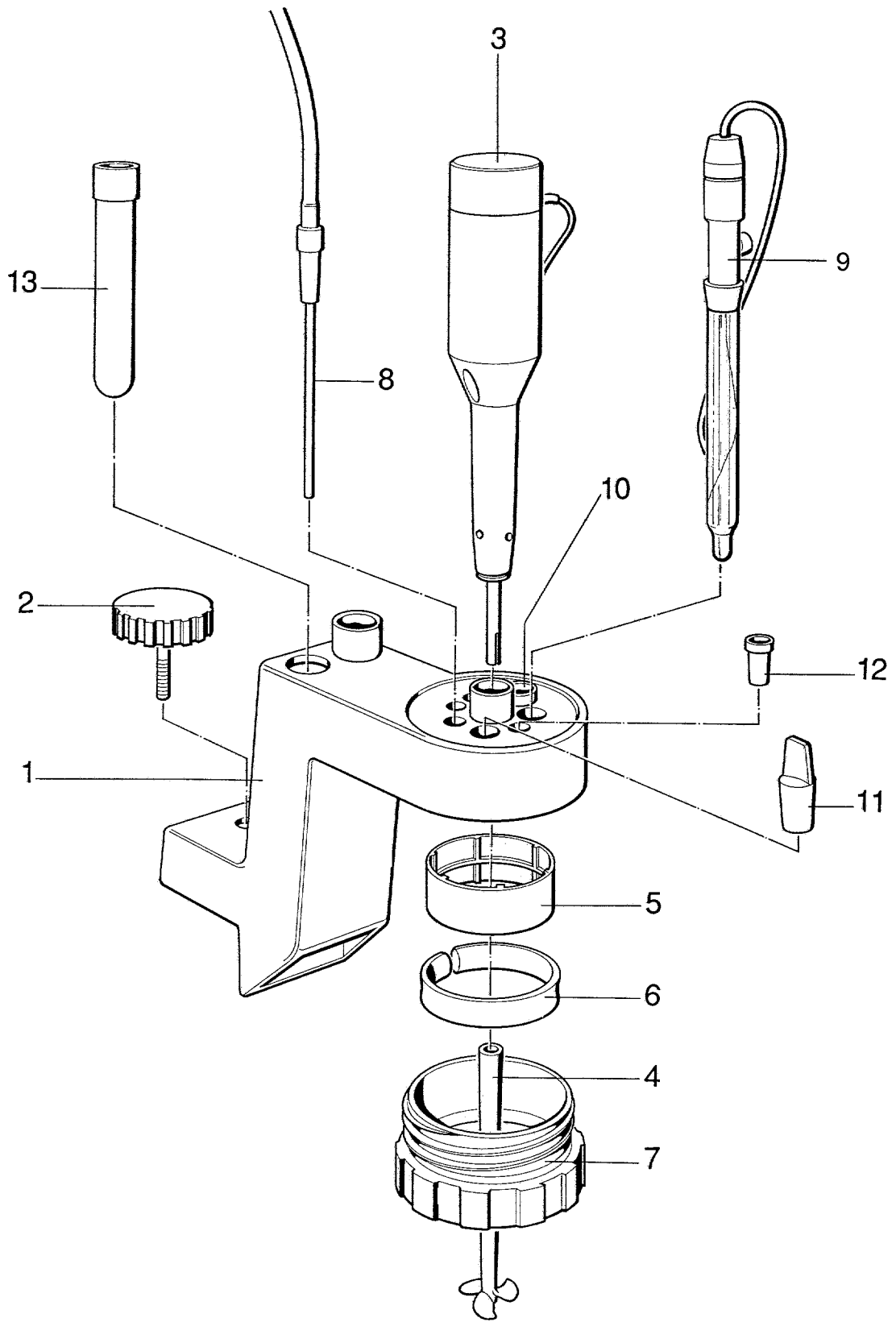
11.1.2.4 General notes

You can remove air bubbles at the piston surface by taking the burette out of the guide and lightly tapping its base. This causes the air bubbles to rise to the top. Then flush the burette.

You can remove air bubbles in the tubes by tapping the tubes with your fingers while titrant is being siphoned off or dispensed. If this does not help, undo the suction tube so that the titrant flows back into the bottle, screw it on again and rinse the burette (air purging). In obstinate cases undo both tubes, rinse with deionized H₂O and ethanol and dry using oil-free compressed air or vacuum.

Titants such as KMnO₄ or KOH in MeOH can easily crystallize in the tip of the burette and block it. If you have no immediate use for a titrant, it is best to empty and clean the dispensing tube: Unscrew connection to let the titrant flow out. Check the threaded connection and wipe off any drops.

KF titrants evolve gas (SO₂) especially at elevated temperatures, causing bubbles to form in the tubing as well as in the burette (stopcock). For this reason burettes should be rinsed prior to titration!



11.1.3 Equipping the titration stand

- Screw on titration arm (1) with knurled screw (2).
- Insert stirring rod (3) and attach propeller stirrer (4) from below to rod.
- Press spacing ring (5) into titration head, place clamping ring (6) in threaded ring (7) and screw in place.
- To install a titration vessel, turn the threaded ring half a turn to the left, attach the vessel and tighten threaded ring.

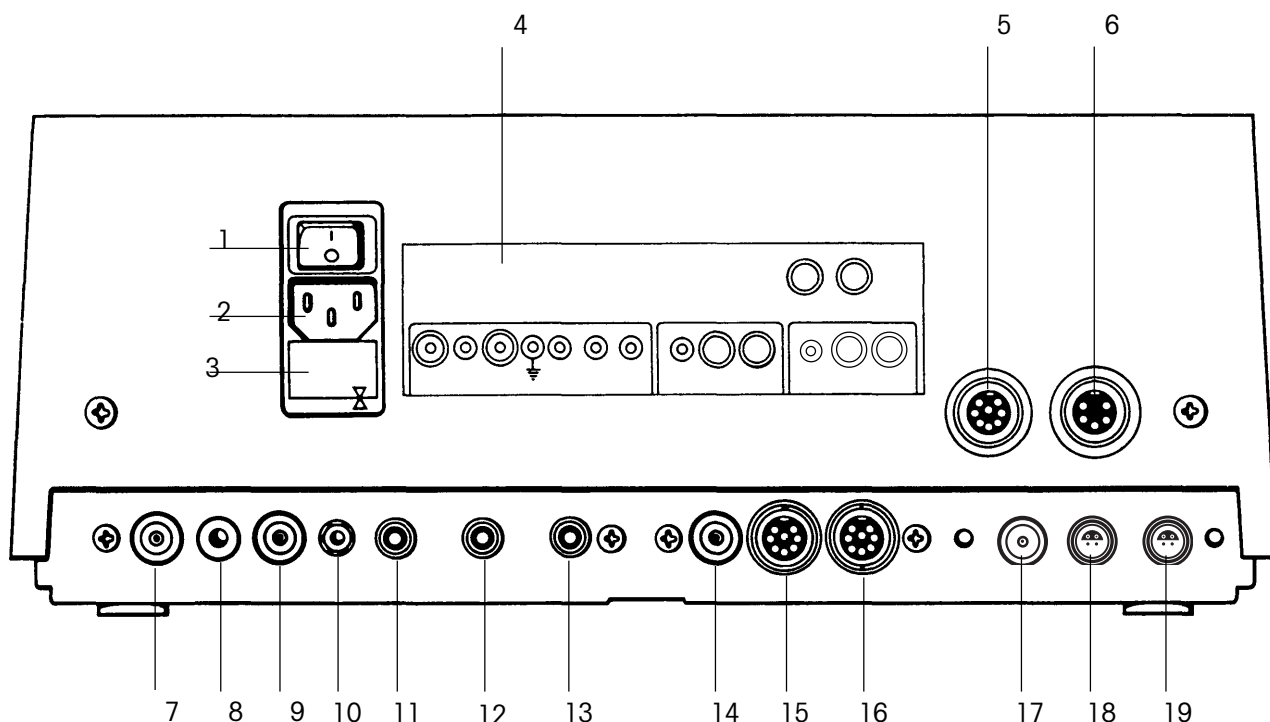


Risk of
corrosion

- Always make sure that the titration vessel is firmly seated in the titration head! When working with toxic solvents, strong acids or bases, you could suffer injury if the titration vessel drops out.

- Insert burette tip (8) and electrode (9) diagonally opposed – this ensures better control in the titration – and close the remaining openings with stoppers (11 & 12).
(10): This opening is intended for the rinsing bottle connection.
(13) is the electrode holder.

11.1.4 Rear view of the titrator



- 1 On/off switch
- 2 Connector for power cable
- 3 Fuse holder and voltage selector
- 4 Sheet with designations of inputs and outputs
- 5 Interface for attachment of a printer (RS232C)
- 6 Interface for attachment of a balance (CL)
- 7 Sensor (electrode) input 1
- 8 Reference electrode input for sensor input 1
- 9 Sensor (electrode) input 2
- 10 Ground socket
- 11 Auxiliary output 1 (for attachment of stirrer, pump, valve or relay)
- 12 Auxiliary output 2 (for attachment of stirrer, pump, valve or relay)
- 13 Auxiliary output 3 (for attachment of stirrer, pump, valve or relay)

RS option (see Section 11.1.6)

- 14 Sensor (electrode) input 3
- 15 Interface for attachment of a Sample Changer (RS232C)
- 16 Interface for attachment of a terminal or computer (RS232C)

Temperature option (see Section 11.1.7)

- 17 Sensor (electrode) input 4
- 18 Temperature sensor input: Temp 1 (Pt1000 or Pt100)
- 19 Temperature sensor input: Temp 2 (Pt1000 or Pt100)

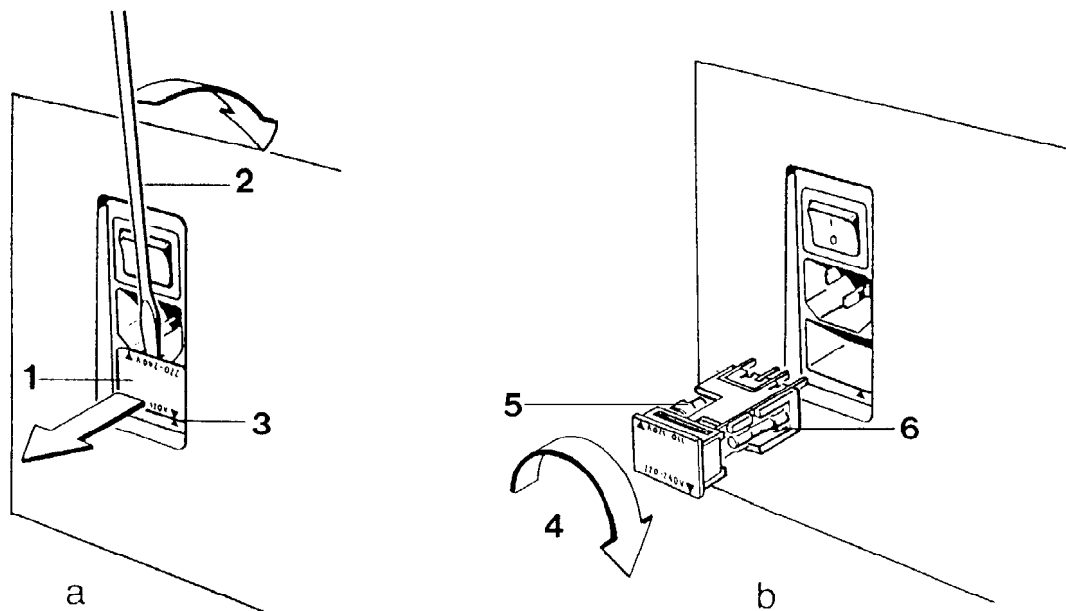
Note Insertion of the two options can also be reversed; the sensor inputs will then be counted from left to right (Sensor 1...Sensor 4).

It is **important** for you to know that

- the sensor input for the RS option is "low resistance",
- the sensor input for the temperature option is high resistance (see Technical data, Section 11.2.1, and Sensors, Section 1.2.2, page 1-11).

11.1.5 Power supply voltage, power fuse

The titrator operates within a voltage range of 100 - 120 or 220 - 240 V. The fuse holder serves as range selector.



Changing the voltage range.

The instrument is set to the voltage range shown by the opposing arrows (3). If you have to change this,

- lever out the fuse holder (1) with a screwdriver (2),
- rotate the fuse holder by 180°(4) and reinsert. This activates the fuse (5).

Replacing defective fuses.

- Lever out the fuse holder (1) with a screwdriver (2).
- Replace the active fuse (6) by one with the same rating.
100 - 120 V: T1.6 L250 V
220 - 240 V: T800L250 V
- Reinsert fuse holder.

Note The standard equipment includes a set of spare fuses for the voltage you specified when ordering the titrator.

Caution The titrator should not be used in a power distribution system without a direct ground connection (IT power system).

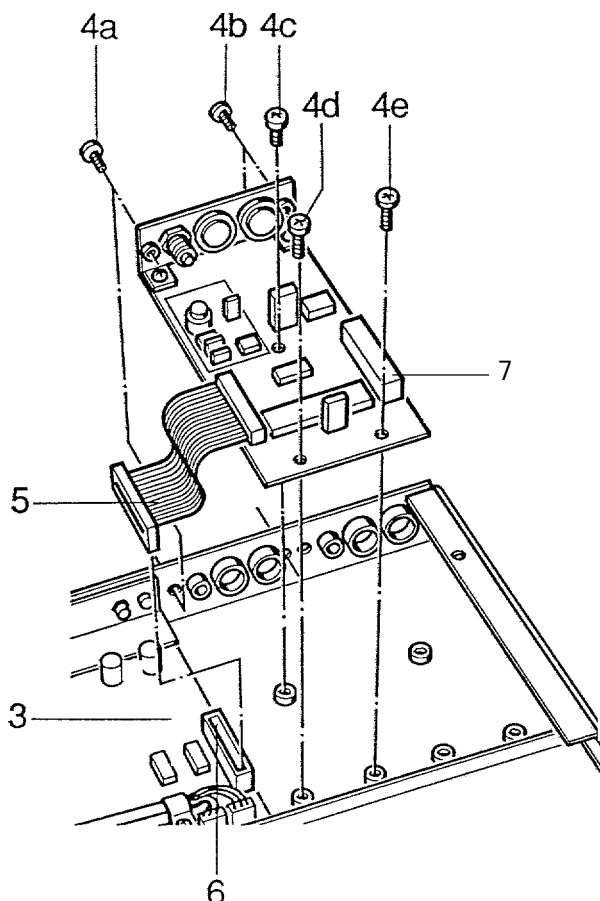
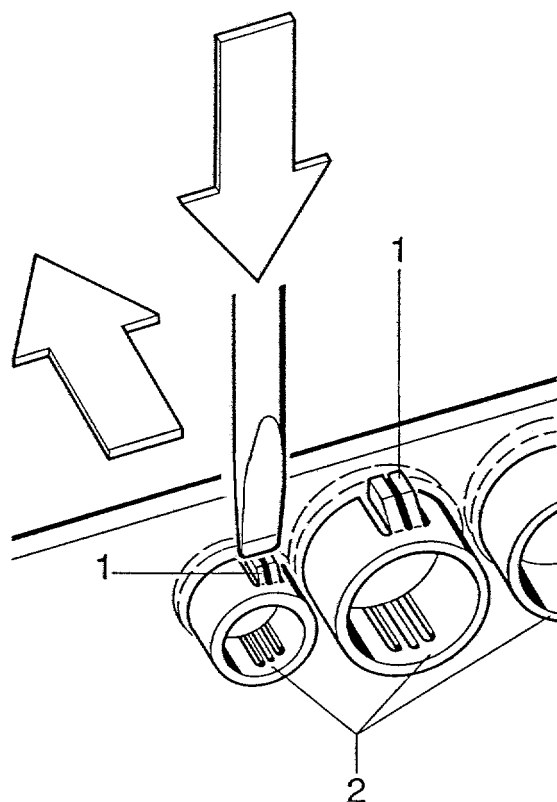
11.1.6 Inserting an RS option (sensor input, 2 RS232C interfaces)

- Slide the burette off the titrator and unscrew the baseplate.



Risk of
electric shock

The titrator must be disconnected from all voltage sources before you remove the baseplate!



This description applies in case you insert the RS option first (see next section).

- Push in protrusions (1) of three plugs (2) with a screwdriver and push out the plugs using your thumbs.
- Place option in housing next to analog board (3).
- First fasten option to housing with two screws (4a & b) then with three screws (4c, d & e).
- Plug connection (5) of cable of RS option into connector (6) of analog board.

(7) is the connector for the next option (see next section).

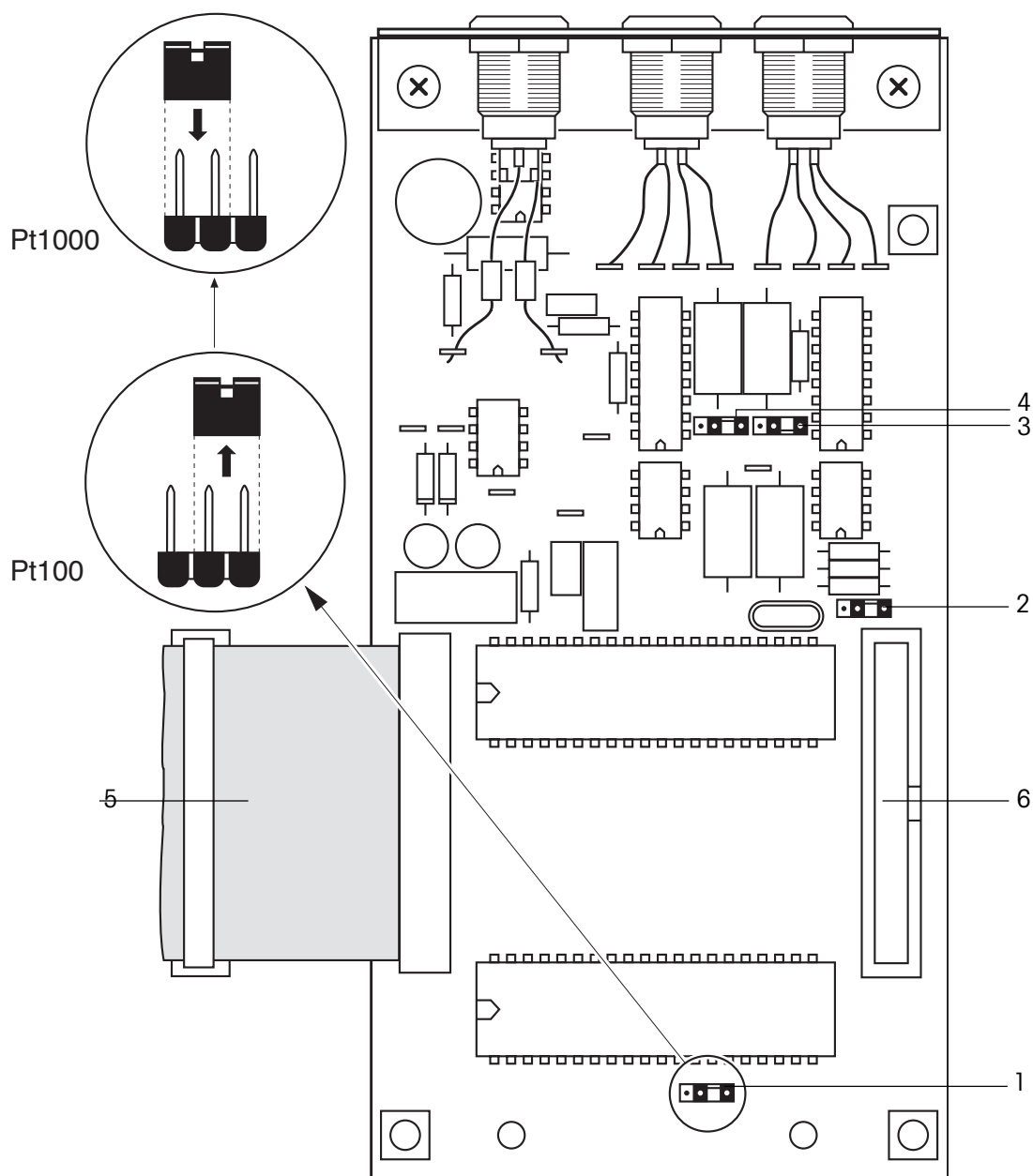
Note When you install this option, you should adjust the sensor input (see Section 4.8: Offset adjustment of the sensor inputs).

11.1.7 Temperature option (1 sensor input, 2 temperature sensor inputs)

Setting

We have set the temperature option to Pt1000 sensors by default.

If you perform temperature measurements with Pt100 sensors, you must alter the setting prior to installing the option.



- Remove all double jumpers on the pin bases (1, 2, 3, 4) and replug one pin to the right of the original position: the option is now set for Pt100 sensors.
(5) is the connection cable, (6) the connector for the next option.

Inserting

Insertion of the temperature option corresponds to that of the RS option (see Section 11.1.6), implying that you can insert either one or the other option.

- Slide burettes off titrator and unscrew the baseplate.



Risk of
electric shock

The titrator must be disconnected from all voltage sources before you remove the baseplate!

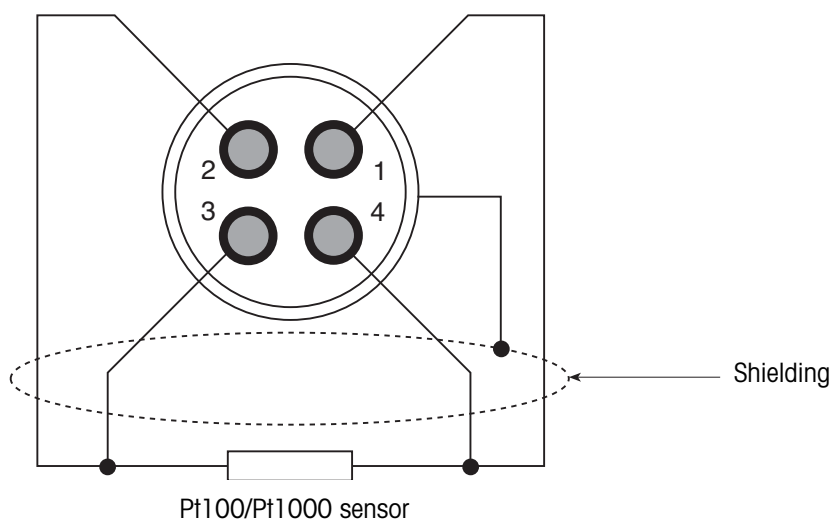
In case you have inserted the RS option already,

- place the temperature option next to it and plug connection of ribbon cable (5) into the connector ((7), see page 11-15) of the RS option.

Notes

- When you install this option, you should adjust the sensor input (see Section 4.8: Offset adjustment of the sensor inputs).
- To connect temperature sensors from other manufacturers, you can obtain a Lemo cable plug (4 pin) and solder on the corresponding cable (see Accessories, Section 11.3.2).

Soldering scheme



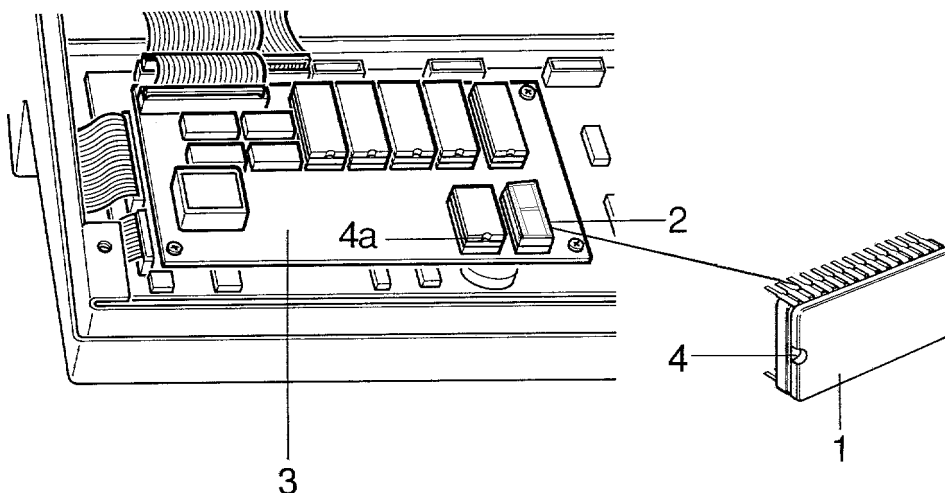
11.1.8 Inserting a user data memory (IC*)



Risk of
electric shock

The titrator must be disconnected from all voltage sources before you remove the baseplate!

- Slide burettes off titrator and unscrew the baseplate.



- Carefully place user data memory (1) on IC socket (2) of processor board (3) with the IC notch (4) facing the same direction as that of the adjacent IC (4a).

* IC is the abbreviation for "Integrated Circuit".

11.2 Technical data

11.2.1 Measurement system

End point indication	Potentiometrically Photometrically (with DP550/DP660) Conductometrically (non Mettler unit with analog output)
Temperature compensation for pH/ pM/ pX measurement	Temperature input via keypad or by means of a temperature sensor
Amplifier range	± 2000 mV
Resolution	0.1 mV, 0.002 pH (pM, pX)
Maximum permissible error	0.1 %
Zero point drift (+10 -> 35 °C)	$< 40 \mu\text{V}/^\circ\text{C}$

Sensor input 1 and sensor input of the temperature option*

Triaxial socket (LEMO)

- Offset current < 1 pA (20 °C)
- Input impedance $> 5 \cdot 10^{12} \Omega$ (protected up to 1000 V capacitively)

Reference electrode input (ref.)

Banana socket 4 mm

- Input impedance $> 3 \cdot 10^7 \Omega$ (protected up to 1000 V capacitively)

Sensor input 2 and sensor input of the RS option*

Triaxial socket (LEMO)

- Offset current < 30 pA (20 °C)
- Input impedance $> 5 \cdot 10^9 \Omega$ (protected up to 1000 V capacitively)

Temperature sensor inputs (Temp 1/Temp 2) of the temperature option

- Range $-20\text{ }^\circ\text{C} \dots 120\text{ }^\circ\text{C}$
- Resolution $0.1\text{ }^\circ\text{C}$
- Maximum permissible error $0.2\text{ }^\circ\text{C}$
- Measurement principle Four terminal resistance measurement: Pt1000 (standard) or Pt100

* The sensor number of the option depends upon its installation (see Sections 11.1.4, 11.1.6 and 11.1.7).

11.2.2 Auxiliary outputs (Aux.1, Aux.2, Aux.3)

Operating modes	Mode 1	For stirrer
	Mode 2	Other auxiliary instruments
Voltage range	Mode 1	0 – 18 V / 150 mA
	Mode 2	24 V / 500 mA

Each output is electronically protected against overload.

11.2.3 Burette drive module

With DC motor

Number burettes	Maximum 4
Resolution	1 / 5000 of burette volume (0.2, 1, 2 and 4 µL)
Maximum permissible error	<0.3% relative to the respective burette volume of 5, 10, and 20 mL
Filling time	20 s
Delivery time	Minimum 20 s

11.2.4 Interchangeable burettes

Number burettes	Maximum 4
Volumes	1, 5, 10 and 20 mL
Materials in contact with titrant	Fluoroplastic, borosilicate glass, ceramics

11.2.5 Propeller stirrer

Maximum speed	No-load operation:	around 4000/min
	In water:	around 3500/min
Power consumption	P _{nominal} :	<4 W
	P _{typical} :	1.2 W at 12 V

11.2.6 Keypad

Material	Polyester , splashproof
----------	-------------------------

11.2.7 Display

Liquid cristal display	with graphics, 6 lines, 36 characters per line, capability 64 * 256 pixels, backlit
Languages	English, German, French, Spanish, Italian

11.2.8 Titration modes

Equivalence point titration	Dynamic or incremental titrant addition Equilibrium controlled or time controlled measured value acquisition Evaluation models for symmetric, asymmetric and segmented titration curves, determination of the minimum or maximum of a titration curve, determination of the half neutralization value.
End point titration	Continuous or incremental titrant addition to absolute or relative end point
Learn titration	
pH-stating	
Karl Fischer titration	Water determination ($>2 \text{ mg H}_2\text{O/sample}$)

11.2.9 Method concept

Flexible sequence through a combination of discrete substages (function) to a method. A particular substage can occur more than once and each is processed in succession.

Functions	Title, Sample, Stir , Measure, Temperature, Instruction, Dispense, Pump, Rinse, Conditioning, Auxiliary instrument, Titration, pH/mV - stat, Calculation, Auxiliary value, Titer , Calibration, Statistics, Record, Sync
Conditional functions	Execution of the functions only if condition is met
Result calculation	Comprehensive calculation of results in the desired unit from all available experimental results, intermediate results and calculated results
Statistical evaluation	Determination of mean value, standard deviation and relative standard deviation, outlier test following Grubbs.
Additional functions	Burette (rinse burette, dispense: fixed volume or continuously , manual titration) Stirrer (stirring and dissolution) Sensor (mV, pH, ... measurement with record of measured values) Temperature ($^{\circ}\text{C}$, $^{\circ}\text{F}$, K measurement with record of measured values) Sample Changer Auxiliary instrument (operation of an auxiliary device; fixed time or manual) Calculations (additional calculations of results at the end of every sample determination) Records (additional record at the end of every sample determination) Calibration of the temperature sensors Offset adjustment of the sensor inputs

11.2.10 Memory

Data base (EPROM)	METTLER methods
User data memory (EEPROM)	Customer methods, installation and analysis data Memory for approx. 50 standard methods

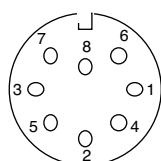
11.2.11 Attachment possibilities for peripherals

Balance	Serial by bit current loop data interface for all METTLER balances with data output CL, 03, 011, 012, 016, 017, 018, 040 A converter cable is available for SARTORIUS balances (CL-RS/RS-CL)
Printer	RS232C interface for various commercial graphics printers with RS232C interface with XON/XOFF protocol.
Sample Changer	Attachment also via RS232C interface (available as option)
Computer	Attachment also via RS232C interface

RS232C interface

Pin assignment of the socket of the titrator and the connectors or sockets of the available cables:

Connection socket of the titrator



Computer (DTE)

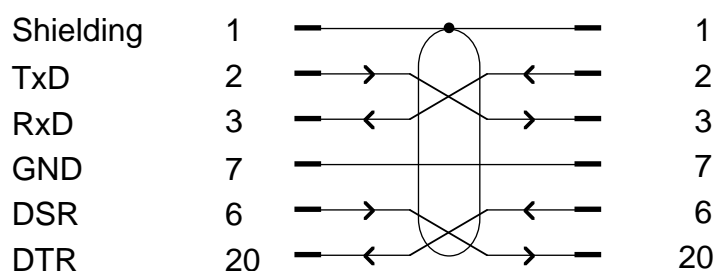
Cables with ME-No. :

			200495 (25 pin, male)	201507 (25 pin, female)	201508 (9 pin, female)
Shielding	1	—	1	1	1
TxD	2	→	2	2	3
RxD	3	←	3	3	2
GND	6	—	7	7	5
DSR	8	→	6	6	6
DTR	4	←	20	20	4
RTS				4	7
CTS				5	8

Null modem

Connector (RS232) for 2 printer cables

Connections D-sub sockets, 25 pin, both female



Additional connections in the connector are allowed.

11.2.12 Additional data

Housing	Polyester
Titration stand	Polypropylene
Dimensions	Width: 360 mm, depth: 400 mm, height: 260 mm
Weight	approx. 11.5 kg with one burette drive
Power supply	100 - 120 V or 220 - 240 V, $\pm 10\%$
Fuse rating	T1,6L250V/ T800L250V
Frequency	50 / 60 Hz
Power consumption	600 mA / 300 mA

Ambient conditions

Ambient temperature	+5 °C ... +40 °C
Atmospheric humidity	Maximum relative atmospheric humidity of 80% for temperatures up to 31 °C, decreasing linearly to 50% relative atmospheric humidity at 40 °C.
Use	indoors
Overvoltage category	II
Pollution degree	2

FCC Rules and Radio Interference Regulations

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to both Part 15 of the FCC Rules and the radio interference regulations of the Canadian Department of Communications. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

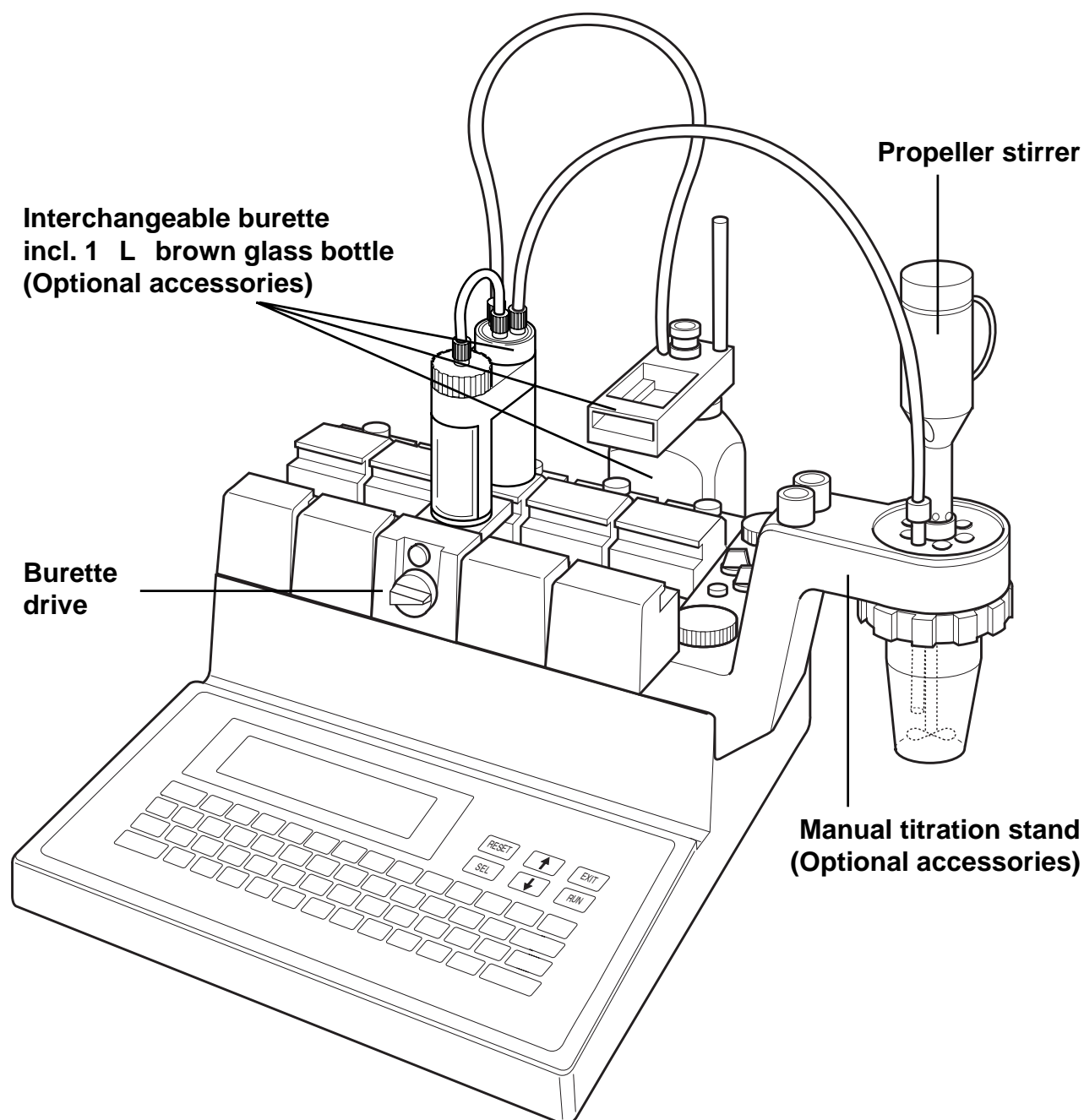
11.3 Accessories

All the instrument's components and working aids for the titrator that form part of

- the **standard equipment** and
- the **optional accessories**

are listed here.

Each part that is listed with an order number can be ordered from METTLER TOLEDO.



11.3.1 Standard equipment

- [1] **DL70ES:** one burette drive¹⁾
DL77: two burette drives¹⁾
- [2] (interchangeable burettes: see Optional accessories)
- [3] One electrode holder
(manual titration stand: see Optional accessories)
- [4] One propeller stirrer incl. 2 stirring rods
- [5] One built-in RS option
- [6] One built-in temperature option
- [7] One serial/parallel converter

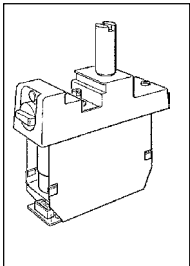
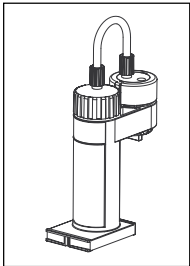

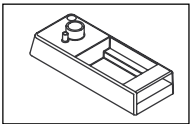
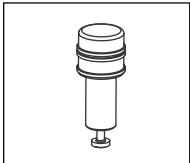
Order No.

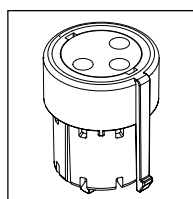
One set of Operating Instructions (DL77/DL70ES/DL67)	in accordance with your order
One Tutorial	in accordance with your order
One RS232C Interface Description	709165
One power cable	in accordance with your order
One set of microfuses	in accordance with your order
One connection cable for a printer	200495
One Phillips screwdriver No. 2	73072
One tube of silicone grease	71300
One electrode cable (SC-LEMO-60)	89601
One short circuit plug	25868
One connection cable for a computer	201508
One CD LabX light titration software	51106330
One set of Operating Instructions (LabX light)	in accordance with your order

¹⁾ As of June 1999, METTLER TOLEDO will no longer manufacture the DL67.

11.3.2 Optional accessories

The numbers enclosed in parentheses refer to the purchase order, e.g., of an interchangeable burette. In case of additional orders, some parts are available only in multipack form or in minimum quantities.

		Order No.	
	[1] Burette drive	DV90	
	[2] Interchangeable burettes		
	Interchangeable burette, complete	1 mL	DV1001
		5 mL	DV1005
		10 mL	DV1010
		20 mL	DV1020
	comprising:		
	1 L brown glass bottle (1)	71296	
	Burette holder	23645	
	Piston (1)	DV1001	51107535
		DV1005	51107115
		DV1010	51107116
		DV1020	51107117

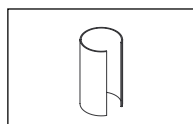
Order No.

Stopcock (1) with valve disk made of
PTFE (light-gray)
ceramic (dark-brown)

51107537

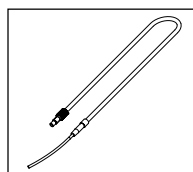
51107525

¹⁾ Both valve disks are chemically resistant. For 24-hour use with titrants that have a tendency to crystallize out, we recommend the ceramic disk.



Light protection tube (1)

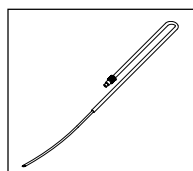
23644



Dispensing tube (1) with siphon tip

70 cm

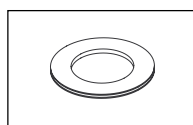
25687



Suction tube (1)

83 cm

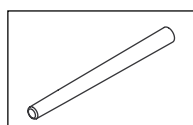
25688



Gasket (1)
for 1 L brown glass bottle

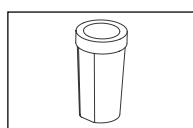
min. order quantity: 5

23981



Tip holder (1) for burette tip

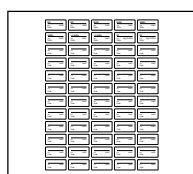
23960



Stopper ST 14.5 (1)

min. order quantity: 5

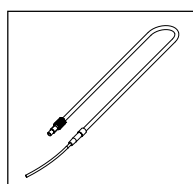
23646



Set of labels (1)

51107506

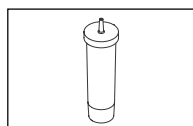
Order No.



Dispensing tube with siphon tip

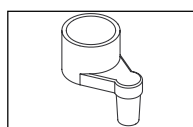
100 cm

25961



Drying tube with cover

23961



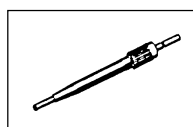
Drying tube holder

23915



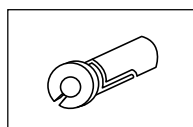
Molecular sieve 250 g

71478



Gas inlet

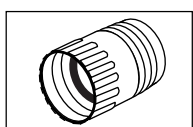
23721



Siphon tip set

set of 5

23240



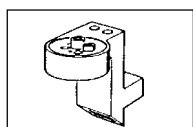
Adapter for bottles by:

Merck, DE

23774

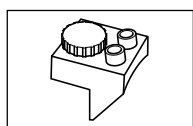
Fisher, US

23787

**[3] Titration stand**

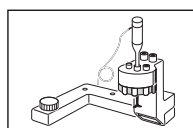
Manual titration stand, complete

51108760



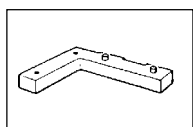
Electrode holder

51108730

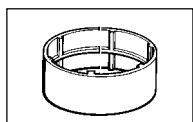
Order No.

Dual titration stand, complete

DV92

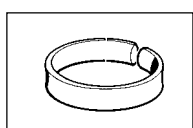
comprising:Propeller stirrer incl. 2 stirring rods (see [4] below) and
Angle bracket (1)

25655



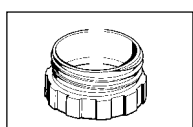
Spacing ring (1)

23842



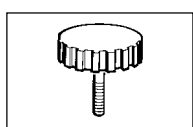
Clamping ring (1)

25653



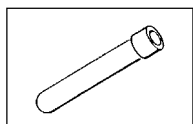
Threaded ring (1)

25652



Knurled screw (2)

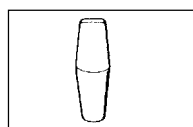
25650



Electrode holder (2)

min. order quantity: 5

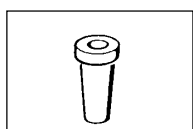
25654



Stopper ST 14.5 (3)

min. order quantity: 5

23451

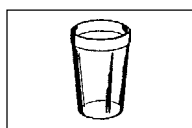


Stopper ST 7.5 (2)

min. order quantity: 5

23452

Order No.

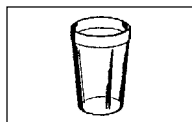


Titration vessel 100 mL
polypropylene (2)

set of 1400

101974

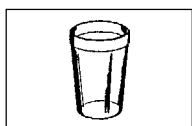
Additional glassware and auxiliary components for the titration stand:



Titration vessel 100 mL
polypropylene, red

set of 1400

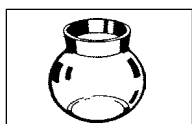
25777



Titration vessel 80 mL, glass

set of 20

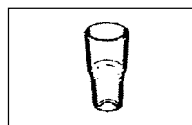
101446



Titration vessel 250 mL, glass

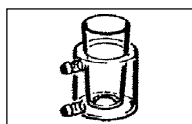
set of 10

23515



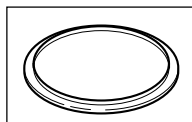
Titration vessel 5-20 mL, glass

23516



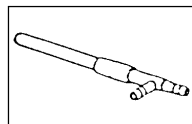
Thermostatable titration vessel 80 mL, glass

23517



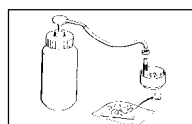
Plastic cover for titration vessels

set of 20 51108481



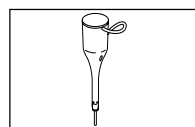
Heat exchanger for thermostating
incl. adapter with taper joint

23834



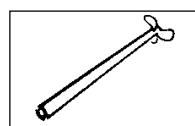
Rinsing unit, complete
with titration head insert and stoppers
for unused openings in titration head
1 set of stoppers for rinsing unit

23821
101230

Order No.**[4] Propeller stirrer**

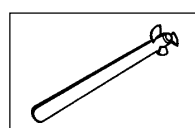
incl. 2 stirring rods

25736



Propeller stirring rod

101229

Micropropeller stirring rod
(for titration vessel 23516)

655073

Caution:Propeller stirrers for the DL40/DL21/DL25 titrators **cannot** be used!**Microfuse**

T1,6 L250V for 100 – 120 V

set of 3

18560

T800L250V for 220 – 240 V

set of 3

20182

**Operating
Instructions**

German

705093

English

705094

French

705095

Tutorial

German

705096

English

705097

French

705098

Spanish

705163

Italian

705164

Memo card

German

709160

English

709161

French

709162

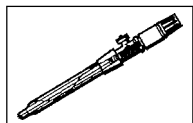
Spanish

709163

Italian

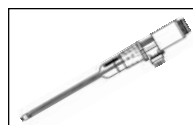
709164

Order No.

Sensors

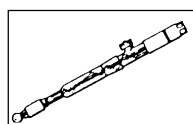
Combined pH electrode for
titrations in aqueous solutions

DG111-SC



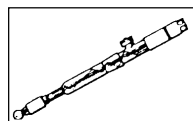
Combined pH electrode for small volumes
in small titration vessels in aqueous solutions

DG101-SC



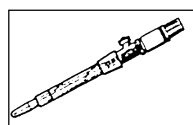
Combined glass electrode with movable sleeve
frit for titrations in nonaqueous solutions

DG113-SC



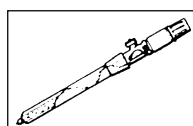
Combined glass electrode with movable sleeve
frit for titrations in aqueous solutions

DG114-SC



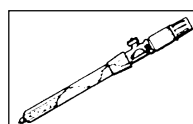
Combined glass electrode with sleeve
frit for titrations in aqueous solutions

DG115-SC



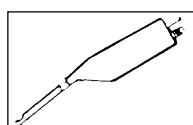
Combined platinum ring electrode for
redox titrations

DM140-SC



Combined silver ring electrode for
argentometric titrations

DM141-SC



Phototrode (incl. power supply unit, cable length 70 cm),
for color-indicated titrations

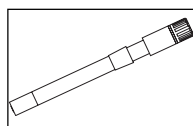
Transmission measurement at 555 nm (green)

DP550

Transmission measurement at 660 nm (red)

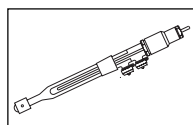
DP660

Order No.



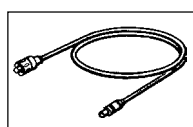
Ion selective measuring electrodes

• Fluoride ISE	DX219	51089931
• Chloride ISE	DX235	51089933
• Nitrate ISE	DX262	51089934
• Sodium ISE	DX223	51089930
• Potassium ISE	DX239	51089932
• Lithium ISE	DX207	51107673
• Ammonia GSE	DX217	51107677
• Ammonium ISE	DX218	51107679
• Magnesium ISE	DX224	51107684
• Cyanide ISE	DX226	51107681
• Sulfide ISE	DX232	51107675
• Calcium ISE	DX240	51107683
• Copper(II) ISE	DX264	51107678
• Bromide ISE	DX280	51107671
• Fluoroborate ISE	DX287	51107676
• Silver ISE	DX308	51107682
• Cadmium ISE	DX312	51107672
• Iodine ISE	DX327	51107680
• Barium ISE	DX337	51107674
• Surfactant sensitive electrode	DS500	51107670



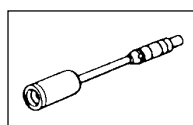
Reference electrode for

• ion selective electrodes	DX200	51089935
• surfactant sensitive electrode	Inlab 301	52000128



Triaxial cable (electrode cable with LEMO connector)

Cable SC-LEMO-60	length 60 cm	89601
Cable SC-LEMO-100	length 100 cm	89602
Cable SC-LEMO-160	length 160 cm	51108034

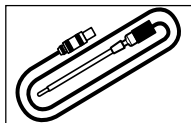


Adapter cable (DIN-LEMO) to attach

electrodes with a DIN connector to the titrator	89600
---	-------

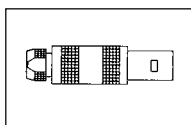
¹⁾ If sensors with DIN connectors are attached to the titrator via the adapter cable, the advantages offered by the triaxial cable – high level of protection against electrostatic interference – are lost in part. When using sensors with a very high resistance, e.g., DG113 in nonaqueous solutions, we advise against use of the adapter cable.

Order No.

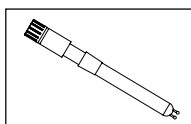
Temperature sensors

Pt1000 sensor

DT1000

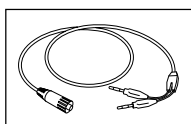
LEMO cable connector (4 pin)
for temperature sensors (non-METTLER)

88321

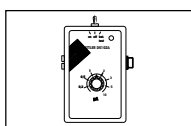
Accessories for Karl-Fischer titrations

Double-pin platinum electrode

DM143-SC

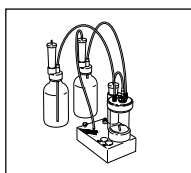
Electrode cable (SC/ Banana)
(Length: 1 m)

51108061



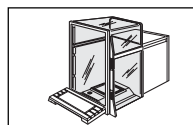
Polarization current source

DK102A

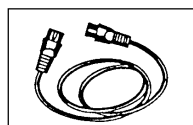


External titration stand

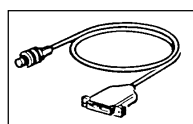
DV705

Order No.**Peripherals**

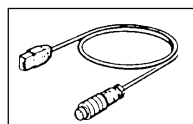
Mettler-Toledo balances with data output AG, AM, PM, AT,
AX, AB, PB, PR



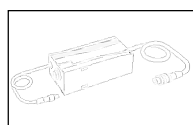
Adapter cable for balances with options 03,
011 and 040 (between option and cable 214101) 42931



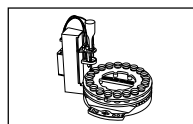
Converter cable for SARTORIUS balances
RS/CL-CL/RS 106024



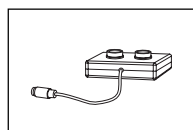
Connection cable for AX, AM, PM, AT balances 214103



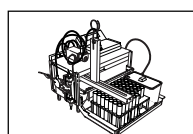
LC-CL cable for AG, AB, PB, PR balances 229130



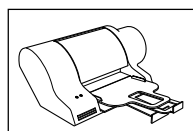
Sample Changer Rondo 60



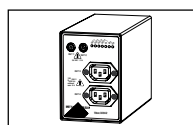
Double cable connecting DL77 with two Rondo 60's 51108305



Sampler system QUANTO aliquot
Sampler system QUANTO direct

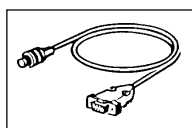


Olivetti Artjet printer
EU version 51190985
US version 51190986



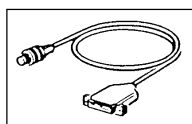
TBox (control of external devices via TTL-I/O) DR42

Order No.



Connection cable (RS232C) for computer
(DTE, 9-pin, female)

201508



Connection cable (RS232C) for computer/terminal
(DTE, 25-pin, female)

201507



LabX professional titration software (incl. Operating Instructions)
English

51106300

German

51106302



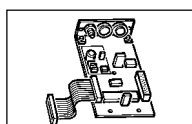
LabX multi titration software (incl. Operating Instructions)

English

51106310

German

51106312

Miscellaneous

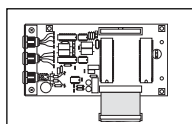
[5] RS option: 2 RS232C (DCE) interfaces
plus 1 sensor input

25690

incl. the Operating Instructions:
"RS232C Description Interface"

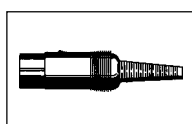
English

709165



[6] Temperature option: 2 inputs for Pt sensors
plus 1 sensor input

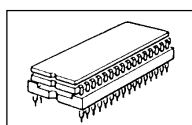
25872



Cable connector 8-pin (DIN)

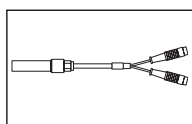
89144

to be prepared as an RS short circuit plug
(for the DL77 to synchronize methods)



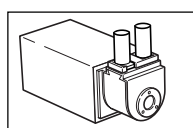
User data memory

25817



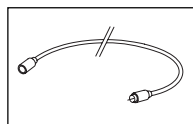
Adapter cable (DIN connector-banana sockets)

25914

Order No.

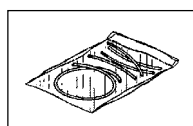
Peristaltic pump SP250
with Novoprene tubes and hose clamps

51108016

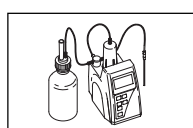


Adapter cable (mini DIN/ RCA)
(Length: 0.5 m)

51108350



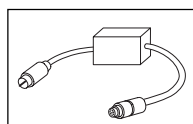
Novoprene tubes for SP250 (1x1 m + 10x120 mm) 51190969
Fluorosilicone elastomer tubes (5x120 mm) 51108149



Dispensing unit DU200
EU version
US version

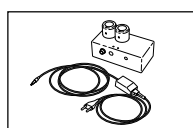
51370200

51370210



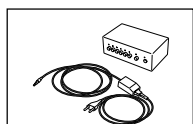
Connection cable DU200 / DL7x

51370511



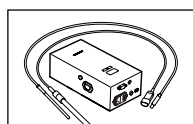
Sampling unit SU24

51108018



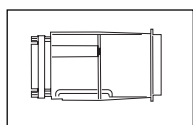
Auxiliary output expander AOE06

51108019



Heating system

DH100

**[7]** Serial/parallel converter

51108024

Order No.**METTLER TOLEDO literature and brochures**

Basics of Titration	51725008
Fundamentals of Titration	704153
Guide to pH Measurement	51300047
Guide to Ion Selective Measurement	51300075
Guide to Conductivity and Dissolved Oxygen	51724716
DL70 Application Brochure 1 (18 Customer methods)	724492
DL70 Application Brochure 2 (Various methods)	724557
DL70 Application Brochure 3 (TAN and TBN)	724559
DL7x Application Brochure 4 (Gold and Silver)	724613
DL7x Application Brochure 5 (Determinations in Water)	51724634
DL7x Application Brochure 6 (Direct Measurement with Ion Selective Electrodes)	51724646
DL7x Application Brochure 7 (Incremental Techniques with Ion Selective Electrodes)	51724648
DL7x Application Brochure 8 (Standardization of Titrants I)	51724650
DL7x Application Brochure 9 (Standardization of Titrants II)	51724652
DL7x Application Brochure 13 (Nitrogen Determination by Kjeldahl Digestion)	51724769
DL7x Application Brochure 14 (Good Laboratory Practice in the Titration Lab)	51724908
DL7x Application Brochure 15 (Guidelines for Result Check, Method Validation and Instrument Certification)	51724910
DL7x Application Brochure 16 (Validation of Titration Methods)	51724912

Index

- ΔE
 - end point titration 2-55
 - equivalence point titration 2-42
 - measure function 2-20
- $\Delta E(\text{set})$
 - end point titration 2-55
 - equivalence point titration 2-40
- Δt
 - end point titration 2-55
 - equivalence point titration 2-42
 - measure function 2-20
 - temperature function 2-22
- Absorption** 1-11
- Accessories**
 - optional accessories 11-28
 - standard equipment 11-27
- ANALYSIS** 3-3
- ANALYSIS A**
 - changing to ANALYSIS B 3-44
 - parallel titrations 3-42
- ANALYSIS B**
 - changing to ANALYSIS A 3-44
 - parallel titrations 3-42
- Analysis parameters**
 - "Installed are" 1-40, 3-8
 - "Results of this sample" 1-40, 3-12
- Applications sheets** 10-15
- Asterisk (*)**
 - method identification 2-5, 2-14
 - previously executed method 3-14
- Asymmetric (evaluation procedure)**
 - equivalence point recognition 2-45
 - evaluation criteria 2-51
 - explanation 8-21
- Audio signal** 1-39
- Automation** 7-7
- Auto stand** 1-25, 3-13
- Aux. 1/2/3 (auxiliary outputs)** 1-18, 1-20, 1-26
- AUXILIARY FUNCTIONS**
 - menu 4-3
 - menu tree 4-4
- Auxiliary instrument(s)**
 - adding 1-21
 - auxiliary function 4-21
 - deleting 1-20
 - function 2-31
 - installing 1-22
 - menu tree 1-21
 - modifying 1-20
- Auxiliary outputs**
 - auxiliary instrument 1-20
 - auxiliary reagents 1-18
 - stirrer 1-26
- Auxiliary reagents**
 - adding 1-18
 - deleting 1-17
 - installing 1-19
 - menu tree 1-19
 - modifying 1-17
- Auxiliary value(s)**
 - entering 1-23
 - entry through determination 1-23
 - function 2-70
 - menu tree 1-24
- Auxiliary value memory** 1-23, 2-70
- Back titration (example)** 2-24
- Balance(s)**
 - configuration (METTLER) 1-30
 - configuration (SARTORIUS) 1-31
 - connection 11-23
 - connection cable 11-37
 - installing 1-30
- Baud rate**
 - balances 1-30, 1-31
 - printer 1-29
 - system 1-32
- Bidirectional transmission mode** 1-30, 3-9
- Buffer potential 1** 2-51, 8-6, 8-9
- Buffer potential 2** 2-51, 8-6, 8-9
- Buffer solutions**
 - DIN/NIST buffer 2-72
 - Ingold buffer 2-73
 - Merck Titrisol buffer 2-73

Burette

- air bubbles 11-9
- equipping 11-7
- inserting 11-8
- installation instructions 11-5
- Karl Fischer titrations 10-5
- maintaining 11-5, 11-8
- menu 4-6
- rinsing 4-6

Burette drive

- inserting 11-3
- selecting 1-5

Burette volume

- selecting 1-5
- smallest increment 2-40

C (constant) 2-67, 8-8

c (nominal concentration) 8-3

c * t (actual concentration) 8-3

Calculation(s)

- designations 8-3, 8-8
- formulae 8-26
- function 2-66
- indexes 8-10
- recalculation 3-34

Calculation operations 2-66**Calibration data**

- determination 2-72
- entry through calibration 1-12
- for sensor inputs 1-12
- theoretical values 1-12

Calibration

- function 2-72
- sensors 2-72
- temperature sensors 4-23

Calibration method 2-74**Character set 1-33****Code (sync fct.) 2-81****Comment (sync fct.) 2-81****Communication protocol 1-33****Communication: titrator <-> computer**

- introduction 7-5
- overview 7-5

Comparison operators 8-17**Computer**

- connection 11-23
- connection cable 11-38
- installing 1-33

Concentration [mol/L] 1-5, 8-3**Conditioning (function) 2-27****Condition for functions 8-16****Conditioning mode**

- fix (explanation) 2-27
- flexible (explanation) 2-27
- selecting 1-26

Conductivity measurement 1-11**Conductometer 1-11****Constants**

- calculation function 2-66
- calculations menu 3-34
- examples 8-27

Continuous (titrant addition) 2-54**Continuous addition (pH/mV-stat fct.) 2-63****Control band 2-54****Control range 2-63****Conversion constant 8-28****Correction factor 3-7****Correlation coefficient 2-62, 2-64****CSTAT (correlation coefficient) 2-62, 8-7, 8-9****Cursor keys**

- during entry 1-4, 2-10
- during titration 3-29

Curve type (display) 3-30**Data administration 7-6****Data backup 7-7****Data bits**

- printer 1-29
- system 1-32

Data entry (LIMS) 3-6**Data storage (pH/mV-stat fct.) 2-64****Data transfer**

- installation data 5-6
- method 5-5

Date

- entering 1-36
- selecting format 1-36

- Date specification
 - auxiliary values 1-23
 - methods 2-14
 - sensors 1-12
 - temperature sensors 1-15
 - titrants 1-6
- Decimal places 2-66, 3-34
- Default speed 1-26, 2-16
- Default values 1-4, 2-10
- Delay 2-54, 2-55
- Designations
 - compilation 8-14
 - explanation 8-3
- Dispense
 - auxiliary function 4-7
 - function 2-24
- Dispenser (sample changer) 4-20
- Dispense continuously 4-8
- Dispensing rate 1-18
- Display (analysis menu) 3-28
- DK102A (polarization current source) 10-6
- DOCUMENTATION 5-3
- DOS (dispense) 2-34
- Dose 1 2-34
- Dose 2 2-34
- Dosing pump (sample changer) 4-19
- Dosing pump manual (sample changer) 4-20
- Drift compensation 10-12
- Drift
 - Karl Fischer titrations 10-7
 - potential measurement 2-20
 - temperature measurement 2-22
- DYN (titrant addition EQP) 2-40
- Dynamic (titrant addition EP) 2-55
- EHN**V (half neutralization value) 2-36, 8-5
- Electrodes (METTLER) 1-9
- E (potential) 2-20, 8-3
- EDITOR
 - menu 2-3
 - menu tree 2-12, 2-13
- End point mode (EP)
 - EPA 2-56
 - EPR 2-56
 - EPS 2-56
- End point mode (pH-stat)
 - EPA 2-63
 - EPS 2-63
- End point range 2-55
- End point titration 2-53
- EP (end point titration) 2-53
- EPA (absolute end point) 2-56, 2-63
- EPOT 2-36, 8-5
- EPR (relative end point) 2-56
- EPS (other end point) 2-56, 2-63
- EQP (equivalence point titration) 2-36
- EQP range 2-49
- EQU (equilibrium controlled measure mode) 2-42
- Equilibrium controlled measured value acquisition 2-42, 2-55
- Equivalence point(s)
 - explanation 2-36, 8-20
 - formulae for limiting 8-31
 - maximum number/method 8-25
- Equivalence point recognition
 - EQP titration 2-45
 - LEARN EQP 2-59
- Equivalence point titration
 - menu tree 2-37
 - titration mode 2-36
- Equivalent number
 - calculation function 2-68
 - entering 2-16
 - modifying 3-7
- Error messages
 - data transfer 5-5
 - learn titration 3-18
 - memory copy 5-9, 5-10
 - remote control 7-3
 - with reference to Section 9.1 9-3
- ET1 2-34, 2-35, 2-39, 2-53, 8-4
- ET2 2-34, 2-39, 2-53, 8-4
- ET3 2-35, 2-39, 8-4

- Evaluation criteria
 - pH/mV-stat function 2-64
 - titration function 2-51
- Evaluation procedure
 - explanation 8-20
 - titration function 2-45, 2-51
- EXIT key 1-6, 2-10, 2-11, 3-8, 3-10, 3-16
- Expert 6-3

- f** (correction factor) 3-7, 8-3
- Fix (conditioning mode) 2-27
- Fixed volume
 - sample data mask 3-10
 - sample function 2-15
- Flexible (conditioning mode) 2-27
- Formulae
 - auxiliary value function 2-70
 - calculation function 2-66
 - calculations menu 3-34
 - constants 8-27
 - for limiting the equivalence point 8-31
 - nominal consumption 8-30
 - nominal content 8-28
 - results 8-26
 - titer function 2-71
- Form feed 1-29
- Frame lines (printout) 1-29
- Free stand 1-25
- Function(s)
 - adding 2-9
 - copying 2-8
 - cutting 2-8
 - deleting 2-8
 - explanation 2-3
 - list 2-3
 - maximum number/method 8-24
 - modifying 2-10, 2-14
 - pasting 2-8
 - selecting 2-8
- Function number 4-3
- Functions with a condition
 - examples 2-79, 8-18, 8-19
 - explanation 8-16
- Fuse (replacement) 11-14

- H** (auxiliary value) 1-23, 2-70, 8-8
- Half neutralization value 2-36
- HELP key 2-39, 2-53, 2-67, 3-34

- ID1 (identification 1)
 - entering 2-16
 - modifying 3-6
- ID2 (identification 2) 3-7
- INC (titrant addition EQP) 2-41
- Indexes 8-10
- Indexing forms
 - compilation 8-14
 - examples 8-10, 8-11, 8-12
- Index key 7
- Inflection point 8-20
- Information
 - enter key: see Tutorial
 - ikey: see Tutorial
 - operating instructions 4
 - system 9-5
- Initial potential 2-34
- INSTALLATION 1-3
- Installation data
 - printing 5-4
- Installation instructions 11-3
- Instruction (function) 2-23
- Interchangeable burettes: see burette
- Interrupting (method) 3-17
- Interval (conditioning fct.) 2-27

- Karl Fischer methods**
 - burette 10-5
 - drift compensation 10-12
 - drift determination 10-7
 - standby titration 10-8
 - titer with Na-tartrate 10-8
 - titration 10-7
- Karl Fischer reagents 10-6
- Karl Fischer titrations
 - performing 10-9
 - limits of detection 10-9
 - reproducibility 10-9
 - results (examples) 10-14
 - safety measures 10-5

- Key combinations
 - compilation 7
- Language 1-37
- Learn titration
 - LEARN EP 2-60
 - LEARN EQP 2-58
 - results 3-13
- Lift (sample changer) 4-16
- Limits ΔV 2-40
- LIMS 7-5
- Line feed 1-29
- List continuous 3-40
- List once 3-37
- Logical operators 8-17
- Loop 2-17
- M** (molar mass) 2-16, 2-68, 3-7, 8-3
- m (weight) 2-15, 3-7, 8-3
- Malfunctions
 - general comments 9-5
 - Karl Fischer titrations 10-13
- Manual
 - auxiliary instrument 4-22
 - temperature entry 2-16, 3-7
- Manual titration
 - interrupting 4-10
 - modifying 4-9
 - starting 4-10
 - terminating 4-10
- Maximum (evaluation procedure)
 - equivalence point recognition 2-45
 - evaluation criteria 2-51
 - explanation 8-23
- Maximum volume
 - end point titration 2-57
 - pH/mV-stat function 2-64
 - termination criterium 2-50
 - titration mode DOS 2-34
- Mean value 2-75
- Measure (function) 2-20
- Measure mode (EQP titration) 2-42
- Measured values
 - displaying 3-29
 - storage 2-64, 8-25
- Measurement points (measured values)
 - for equivalence point recognition 2-45, 2-59
 - maximum number/titration fct. 8-25
- Memory copy
 - computer/titrator 5-7
 - titrator 1/titrator 2 5-9
 - titrator/computer 5-8
- Menu (explanation) 4
- Menu change
 - with key combinations 7
- Menu sequences
 - example for sample data entry 3-11
 - method M001 3-4, 3-5
- Menu tree (explanation) 4
- Menu trees
 - auxiliary functions 4-4
 - auxiliary instruments 1-21
 - auxiliary reagents 1-19
 - auxiliary values 1-24
 - editor menu 2-12, 2-13
 - equivalence point titration 2-37
 - overview (DL77) 5
 - sensors 1-13
 - system with computer 1-34
 - temperature sensors 1-16
 - titrants 1-7
 - titration function 2-33
 - titration stands 1-27
- Method (ANALYSIS)
 - adding 3-6
 - cutting 3-36
 - deleting 3-36
 - executing 3-4
 - fading out 3-16
 - interrupting 3-17
 - loading 3-6, 3-8
 - modifying 3-21
 - pasting 3-36
 - recording 3-32
 - restarting 3-14
 - starting 3-8
 - terminating 3-15
 - time acquisition 3-13

- Method (EDITOR)
 - copying 2-7
 - deleting 2-7
 - explanation 2-3
 - modifying 2-7
 - printing 2-6
 - recording 2-77
 - saving 2-11
 - selecting 2-5
- Method (selection menu)
 - method completed 3-13
 - method faded out 3-16
 - method interrupted 3-17, 3-19
 - starting method 3-8
- Method data
 - menu 3-26
 - modifying (before starting method) 3-6, 3-26
 - modifying (during current method) 3-26
 - printing 3-27
- Method data list 3-26
- Method data mask
 - notes 3-10
- Methods
 - development (design scheme) 8-36
 - examples 8-32
 - maximum number/method list 8-25
 - printing (list) 5-3
- Method ID
 - entering 2-14, 3-6
 - explanation 2-5
 - modifying 2-11
 - overwriting 2-11
- Method list
 - filling 3-35
 - modifying 3-36
 - processing 3-37
- Method series 2-26
 - with 2 sample changers 3-46
 - titration sequences 3-38, 3-39
- METTLER methods
 - list 10-4
 - memory 11-22
 - selecting 2-5
- Minimum (evaluation procedure)
 - equivalence point recognition 2-45
 - evaluation criteria 2-51
 - explanation 8-23
- Miscellaneous (menu) 1-36
- Moisture determination 10-5
- Molar mass
 - calculation function 2-68
 - entering 2-16
 - modifying 3-7
- neq (number of equivalence points) 8-5
- New method 2-6
- Nominal content
 - dispensing 8-28
 - predispensing 8-28
- Null modem
 - pin assignment 11-24
- Number samples
 - entering 2-15
 - modifying 3-6
- Numeric keys 4-3
- Offset adjustment 4-24
- Operating concept 4
- Outlier test 2-75
- Output unit 2-77
- P1/P2 8-6, 8-9
- Paper (printer)
 - fanfold 1-28
 - single sheet 1-28
- Paper format 1-29
- Parallel titrations
 - notes 3-43
 - procedure 3-42
- Parity
 - balances 1-30
 - printer 1-29
 - system 1-32
- Peripherals (menu) 1-28
- pH-stating (example) 2-65
- pH/mV-stat (function) 2-62
- pH measurement 4-12

- Piston
 - assembling 11-5
 - inserting 11-5
- Potential measurement 4-12
- Power fuse 11-14
- Power supply voltage 11-14
- Predispensing 1 2-39
- Predispensing 2 2-39
- Predispensing
 - end point titration 2-53
 - equivalence point titration 2-39
- Pretitration 2-63
- Printer
 - connection 11-23
 - connection cable 11-27
 - Diabolo 1-28
 - DICONIX 180si 1-28
 - HP Deskjet 1-28
 - IBM 1-28
 - installing 1-28
 - LX800 1-28
- Pt100 sensor 1-15
- Pt1000 sensor 1-15
- Pump (function) 2-25

- Q** 2-36, 8-5
- QDISP 2-24, 8-3
- QEX 8-5
- QP1/QP2 8-6, 8-9
- QSTAT 2-62, 8-8
- QT 8-7, 8-9
- QT1/QT2 2-62, 8-7, 8-9
- QTOT 2-62, 8-7
- Question mark (?) 2-5, 2-14

- R (result)** 2-66, 8-8
- Raw results
 - compilation 8-9
 - explanation 2-21
 - recording 2-78, 3-32, 8-9
 - storage 8-25
- Rear view of the titrator 11-12
- Recalculation 3-34
- Recommendation menu 1-5
- Record (function) 2-77
- Records (analysis menu) 3-32
- Record header 1-37
- Reevaluation 2-52, 3-19
- Reference electrode 1-9
- Relative standard deviation 2-75
- REMOTE CONTROL 7-3
- Remote control 7-7
- Representation (display) 3-29
- RESET
 - auxiliary functions 4-5
 - current method 3-15
 - documentation 5-3
 - list continuous 3-40
- Resources
 - deleting 1-3
 - explanation 1-3
 - list 1-3
- Result(s)
 - displaying 3-29
 - examples 2-67, 2-68, 8-26
 - Karl Fischer titrations 10-14
 - learn titration 3-13
 - maximum number/method 8-25
 - R 2-66, 2-69
 - recording 2-78, 3-32
 - Ri 2-75
 - sample data mask 3-10
 - storage 8-25
 - units 2-67, 2-68, 8-26
- Result list 3-13
- Result units 2-66, 3-34
- Rinse
 - conditioning function 2-27
 - function 2-26
- Rinse tip 4-7
- Rinsing pump (sample changer) 4-18
- Rinsing pump manual (sample changer) 4-18
- Routine 6-3
- Routine level 1-38
- RS232C interface
 - pin assignment 11-23
- RS option
 - accessories 11-38
 - inserting 11-15

- s** (standard deviation) 2-75, 8-8
- Safety measures
 - for your protection 1, 10-5, 11-3, 11-11
 - for operational reliability 1
- Sample (function) 2-15
 - responsibility 2-16
- Sample changer
 - auxiliary function 4-16
 - connection 11-23
 - connection cable 11-38
 - connection scheme 1-35
 - installing 1-35
 - method series 3-46
 - sample series 3-45
 - titration sequences 3-38, 3-39
- Sample data
 - entering (before starting method) 3-7, 3-23
 - entering (during current method) 3-10, 3-23
 - entering (for "List continuous") 3-41
 - menu 3-23
 - printing 3-27
 - recording 2-77, 3-32
- Sample data list 3-23, 3-25
- Sample data mask
 - notes 3-10
- Samples
 - maximum number 8-24
- Sample series
 - example 2-17
 - sample function 2-16
 - statistics function 2-17
 - with 2 sample changers 3-45
- Save
 - measured values (pH/mV-stat fct.) 2-64
 - methods 2-11
 - modified "current" method 3-21
 - parameters 2-10
 - reevaluation 3-20
 - resources 1-3, 1-6
 - with key combinations 7
- Scheme (method design) 8-36
- Segmented (evaluation procedure)
 - equivalence point recognition 2-45
 - evaluation criteria 2-51
 - explanation 8-22
- SEL key 1-5, 2-10
- Selection menu 1-5
- Send mode 1-33
- Sensor(s)
 - adding 1-14
 - auxiliary function 4-12
 - deleting 1-10
 - installing 1-14
 - menu tree 1-13
 - modifying 1-10
 - overview 1-9
- Sensor input(s)
 - adjusting 4-24
 - calibration data 1-12
 - for glass electrodes 1-9, 1-11
 - for phototrodes 1-9, 1-11
 - for temperature sensors 1-15
 - RS option 1-11
 - temperature option 1-11
- Series: see sample or method series resp.
- Single method 3-37
- Slope (sensor)
 - calculation 2-72
 - entry through calibration 1-12
 - explanation 1-12
 - pH calibration 2-73
 - theoretical value 1-12
- Software version 4
- Speed
 - entering 1-26, 2-18
 - modifying 3-31
- srel (rel. standard deviation) 2-75, 8-8
- ST20A (ST20) sample changer 1-35
- ST20 1/ST20 2 1-25
- Standard (evaluation procedure)
 - equivalence point recognition 2-45
 - evaluation criteria 2-51
 - explanation 8-20
- Stand 1/2 1-25
- Standard deviation 2-75
- Start/end character 1-33
- Statistics (function) 2-75

- Status
 - remote control 7-3
 - stirrer 3-31
- Steepest jump only 2-51
- Stir (function) 2-18
- Stirrer
 - analysis menu 3-31
 - auxiliary function 4-11
- Stirrer connection 1-26
- Stop bits
 - printer 1-29
 - system 1-32
- Stop for reevaluation 2-52
- Submethods 8-34
- Sync (function) 2-81
- Synchronization mode 2-81
- System 1-32

- T** (temperature) 2-22, 8-3
- t (titer) 2-71, 8-8
- t(max)
 - end point titration 2-55
 - equivalence point titration 2-42
 - measure function 2-21
 - pH/mV-stat function 2-64
 - temperature function 2-22
- t(min)
 - end point titration 2-55
 - equivalence point titration 2-42
 - measure function 2-21
 - pH/mV-stat function 2-64
 - temperature function 2-22
- Table of measured values
 - displaying 3-29
 - recording 2-78, 3-32
- Technical data
 - auxiliary outputs 11-20
 - burettes 11-20
 - burette drive module 11-20
 - display 11-21
 - general data 11-24
 - keypad 11-21
 - measurement system 11-19
 - memory 11-22
 - propeller stirrer 11-20
- Temperature
 - auxiliary function 4-14
 - entering 1-12, 3-7
 - entry through calibration 1-12
 - function 2-22
 - measuring 1-12, 2-16, 2-20, 2-22, 2-32, 2-62, 2-72, 3-7, 4-14
- Temperature compensation 2-16, 2-72
- Temperature option
 - accessories 11-38
 - inserting 11-17
 - setting 11-16
- Temperature sensor(s)
 - calibrating 4-23
 - Lemo cable plug 11-17
 - menu tree 1-16
 - modifying 3-7
 - selecting 2-16
 - Pt100 1-15
 - Pt1000 1-15
- Tendency
 - end point titration 2-57
 - EQP range 2-49
 - LEARN EP 2-61
 - pH/mV-stat function 2-63
- Terminal
 - configuration 7-9
 - connection 11-23
 - installing 1-32
 - key assignment 7-10
- Terminate: see RESET
- Termination
 - after n EQP's 2-50
 - after nominal consumption (example) 8-30
 - after record 2-79
 - at combined criteria 2-51
 - at maximum volume 2-50
 - at potential 2-50
 - at slope 2-50
- Termination criteria
 - pH/mV-stat function 2-64
 - titration function 2-50
- TFIX (time increment measure mode) 2-44
- Threshold 2-45

- Threshold value
 - evaluation procedure minimum 2-46
 - evaluation procedure segmented 2-48
 - evaluation procedure standard/asymmetric 2-47
- TIME 3-13, 8-8, 10-12
- Time-controlled (auxiliary instrument) 4-21
- Time acquisition (method) 3-13
- Time
 - auxiliary instrument function 2-31
 - conditioning function 2-27
 - entering 1-36
 - selecting format 1-36
 - stir function 2-18
- Time interval 2-64
- Time limits 2-64
- Time span 2-64
- Time specification
 - auxiliary values 1-23
 - methods 2-14
 - sensors 1-12
 - temperature sensors 1-15
 - titrants 1-6
- Titer
 - determination (method excerpt) 2-71
 - entering 1-5
 - entry through titer determination 1-5
 - function 2-71
- Title (function) 2-14
- Title line (parameter mask) 1-6, 2-10
- Titration
 - function 2-32
 - menu tree 2-33
- Titration curves
 - 1. derivative 2-47, 2-48, 8-22
 - 2. derivative 2-48, 8-22
 - displaying 3-29
 - recording 2-78, 3-32
- Titration mode 2-32
- Titration sequences
 - comparison (stand 1/ST20 1) 3-38
 - excerpt of a standard method 3-12
 - on the sample changer 3-39
 - pH-stating 3-24
- Titration stand(s)
 - equipping 11-11
 - importance 2-16
 - installation data 1-25
 - menu tree 1-27
 - modifying 3-7
 - selecting 2-15
- Titration time 3-13, 8-8, 10-12
- Titration ID 1-37
- Transmission 1-11
- Transmission mode
 - bidirectional 1-30
 - unidirectional 1-30
- Turntable (sample changer)
 - backward 4-17
 - forward 4-17
- U** (volume) 2-15, 8-3
- Unidirectional transmission mode 1-30
- Units 2-67, 8-27
- Units of measurement
 - potential 1-10, 1-11
 - temperature 2-22
- User data memory
 - accessories 11-39
 - inserting 11-18
 - technical data 11-22
- USER LEVEL 6-3
- User methods 2-6

VDISP 2-24, 8-3

VEQ 2-36, 8-5

VEX 8-5

Voltage range 11-14

Volume

entering 3-7

selecting 2-15

Volume limits

at volume entry 3-9

entering 2-15

VP1/VP2 8-6, 8-9

VSTAT 2-62, 8-8

VT 2-64, 8-7, 8-9

VT1/VT2 2-62, 8-7, 8-9

VTOT 2-62, 8-7

Water determination 10-5

Weight

entering 3-7

selecting 2-15

transferred by balance 3-9

Weight limits

at weight entry 3-9

entering 2-15

in the sample data mask 3-7

\bar{x} (mean value) 2-75, 8-8

z (equivalent number) 2-16, 2-68, 3-7, 8-3

Zero point (sensor)

calculation 2-72

entry through calibration 1-12

explanation 1-11

theoretical value 1-12

Zero point (temperature sensor)

entry through calibration 1-15

explanation 1-15

theoretical value 1-15

ISO 9001 certificate for METTLER TOLEDO

The Mettler-Toledo GmbH company, Greifensee, was examined and evaluated in 1991 by the Swiss Association for Quality and Management Systems (SQS), and was awarded the ISO 9001 certificate. This certifies that Mettler-Toledo GmbH, Greifensee, has a quality management system that conforms with the international standards of the ISO 9000 series.

Repeat audits are carried out by the SQS at intervals to check that the quality management system is operated in the proper manner and is continuously updated in relation to changes brought about.

Certificado ISO 9001 para METTLER TOLEDO

La firma Mettler-Toledo GmbH, Greifensee, ha sido inspeccionada por la Asociación Suiza para Sistemas de Calidad y Gestión (SQS), habiendo obtenido el certificado ISO 9001. Esto acredita que Mettler-Toledo GmbH, Greifensee, dispone de un sistema de gestión de calidad que cumple las normas internacionales (ISO serie 9000).

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Certificato ISO 9001 per la METTLER TOLEDO

Il sistema di garanzia della qualità della Società Mettler-Toledo GmbH, Greifensee, è certificato ISO 9001 sin dal 1991 dall'Associazione Svizzera per Sistemi di Qualità e di Gestione (SQS), e così fornisce la dimostrazione che il suo sistema Garanzia di Qualità soddisfa i massimi requisiti.

Il sistema della garanzia della qualità Mettler-Toledo viene verificato periodicamente SQS, dando così evidenza di un continuo aggiornamento e corretta gestione.

Declaration of System Validation

We herewith inform you that the products/systems:

DL67, DL70ES, DL77

including software and accessories were developed, tested and successfully validated according to the international ISO9001:1994 based Life cycle rules of Mettler-Toledo GmbH, Analytical.

Life cycle checkpoint details were reviewed and approved by the Project Supervisory Group (PSG). The products/systems were tested to meet functional and performance specifications and release criteria at release to shipment. In order to support GLP and validation requirements, we will make the following documents available to an authorized, governmental or regulatory agency for inspection.

- Product Guidelines
- Performance Specifications
- Documentation Plan
- Software Specifications
- Quality Plan
- Project Management System
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- Customer Requirements
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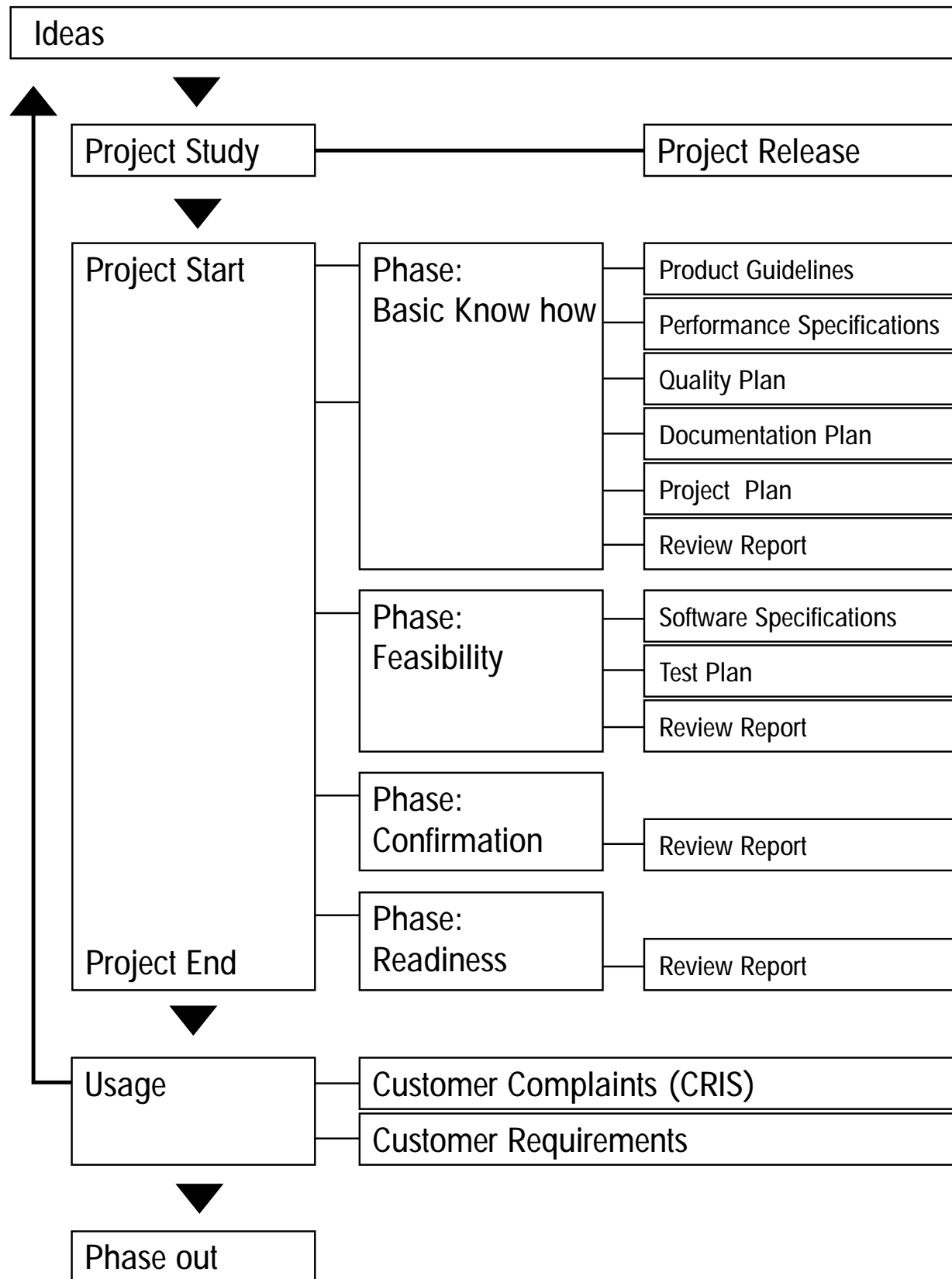
Schwerzenbach,
March 1999

Dr. Bernhard Grob
General Manager
Business Unit Analytical



Dr. Urs Spitz
Manager Business Area
Titration

Product Life Cycle Model



Déclaración de validación del sistema

Por la presente le informamos que los productos/sistemas

DL67, DL70ES, DL77

incluido el software y los accesorios, han sido desarrollados, probados y verificados con éxito de acuerdo con las reglas sobre ciclo de vida de producto de Mettler-Toledo GmbH, Analytical. Estas reglas se basan en la norma 9001:1994.

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- Imagen del producto
- Especificaciones
- Documentación del proyecto
- Especificaciones del software
- Plan de calidad
- Directiva Gestión de Proyecto
- Plan de ensayos
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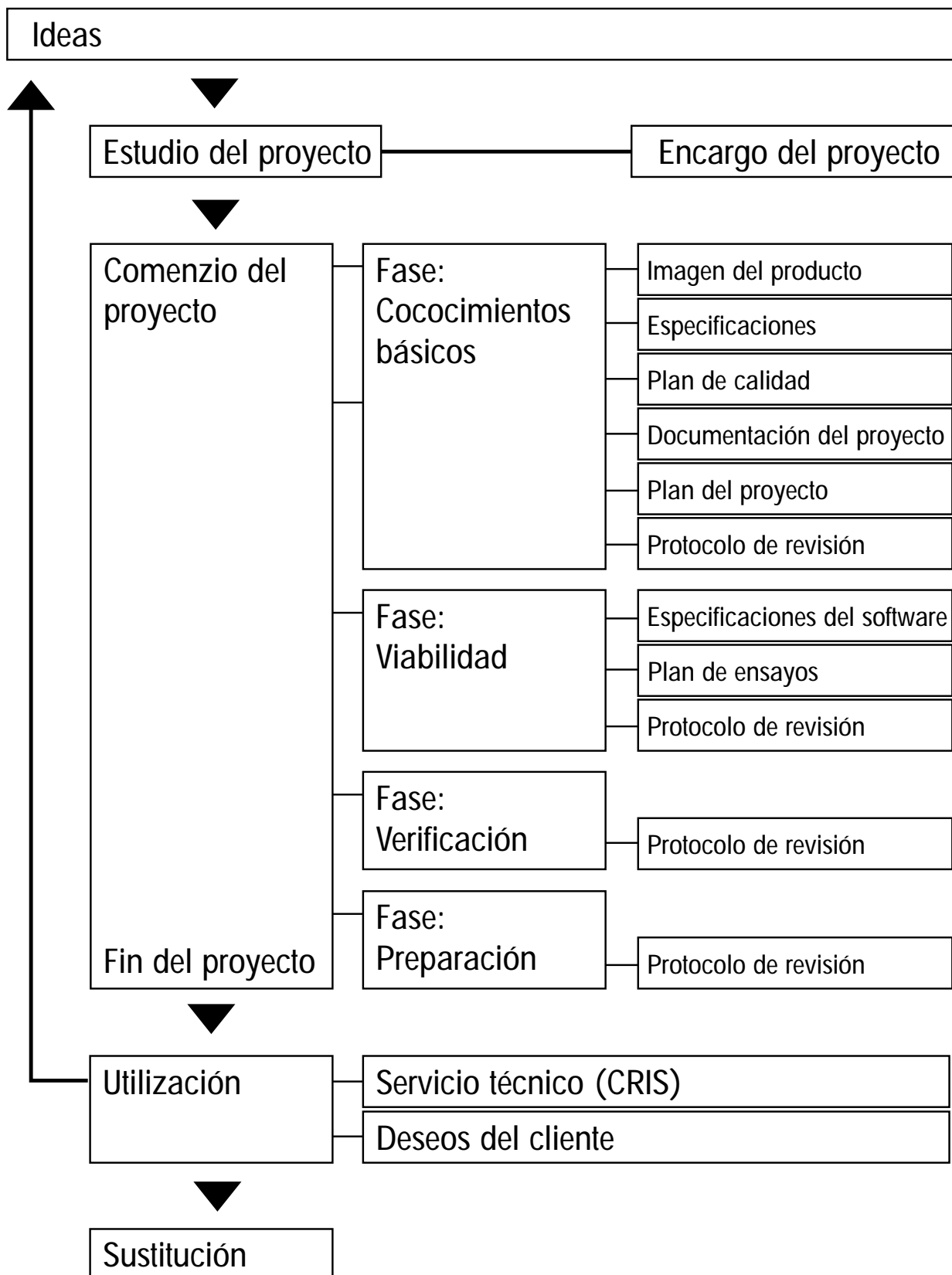
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Dr. Bernhard Grob
General Manager
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Dr. Urs Spitz
Manager Business Area
Titration

Modelo del ciclo de vida de un producto



Dichiarazione di validazione del sistema

Con la presente Vi informiamo che i prodotti/sistemi

DL67, DL70ES, DL77

inclusi software e accessori sono stati sviluppati, controllati e validati secondo le regole tecniche relative al ciclo vita dei prodotti della Mettler-Toledo GmbH, Analytical. Queste regole si basano sulle norme ISO 9001:1994.

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- Specifiche del prodotto
- Linee direttive
- Documentazione del progetto
- Specifiche del software
- Sistema di qualità
- Disposizioni per la gestione del progetto
- Piano dei test
- Dati del servizio clientela/esigenze dei clienti
- Rapporti di revisione
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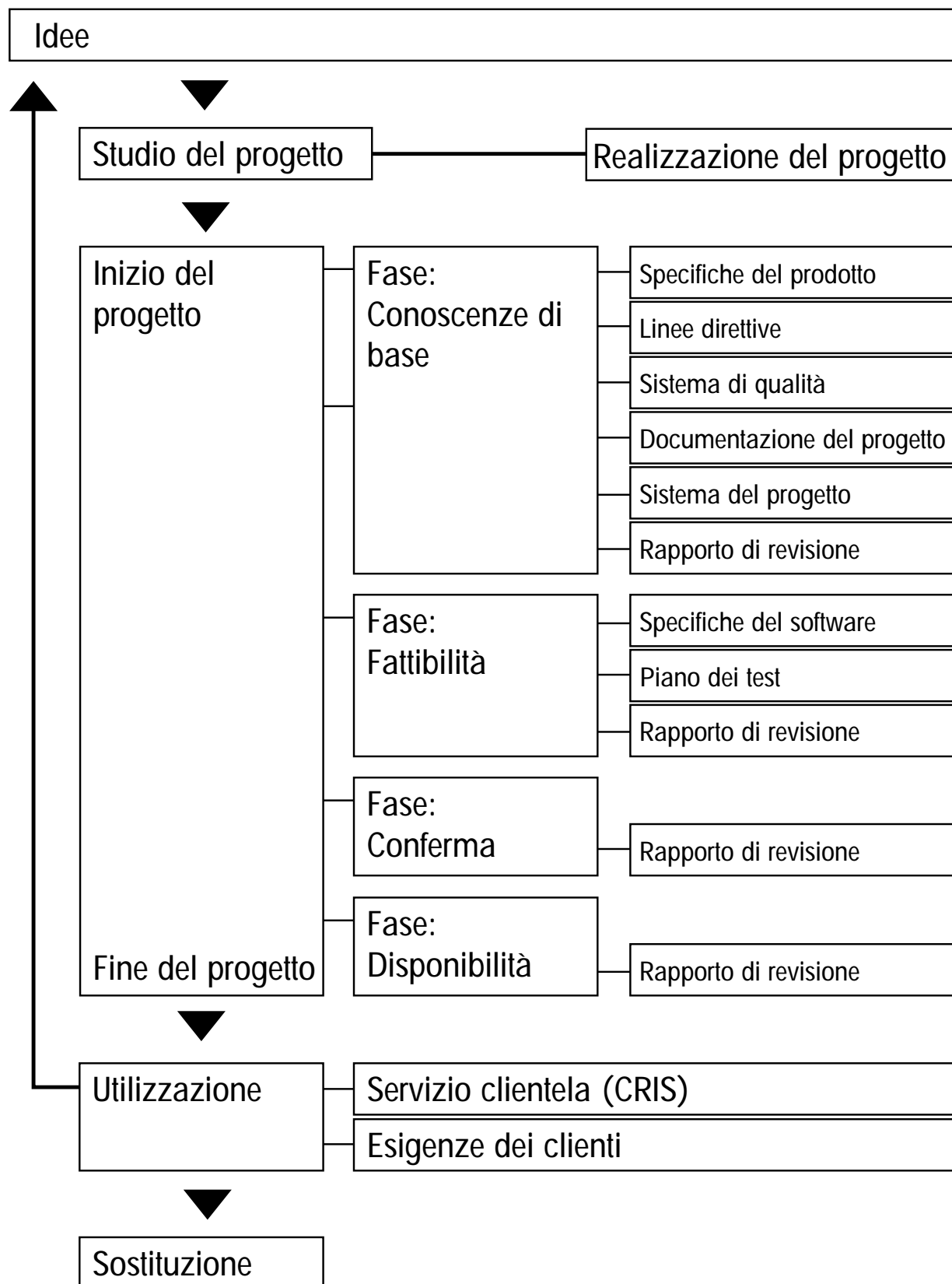
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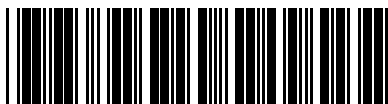
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Modello di ciclo vita di un prodotto



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